# AMERICAN SOCIETY
# *for*
# TESTING MATERIALS

(Reg. U. S. Pat. Office)

## PROCEEDINGS

VOLUME 57
1957

COMMITTEE REPORTS
TECHNICAL PAPERS

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# FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings*, together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* include those accepted for publication in the ASTM BULLETIN or in Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1957 is given on page 1409 of this volume. This supplements the lists appearing in the *Proceedings* from 1948 to 1956 covering all special publications published by the Society up to and including 1956.

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# SUMMARY OF PROCEEDINGS
OF THE
# SIXTIETH ANNUAL MEETING

Atlantic City, N. J., June 17–21, 1957

This summary of the Sixtieth Annual Meeting of the American Society for Testing Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 16–21, 1957, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 31 technical sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 1628; committee members, 935; guests, 369; total, 2932; ladies, 140.

The Proceedings are set forth session by session. There were 65 reports and 79 formal papers presented. The record with respect to each has been briefed; the recommendations in the reports have been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in," it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society.[1] The various recommendations so recorded are included in the Society letter ballot. The actions designated as "accepted as tentative" or "accepted as tentative, revisions in" are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative. Designations that have since been assigned to new tentatives are included as information.

All papers, unless otherwise noted, are published in the Proceedings.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded. There are also listed the various symposia and special sessions.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, will be canvassed on September 16, 1957. See Editors' Note, p. 32.

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## FIRST SESSION—OPENING SESSION

MONDAY, JUNE 17, 10:00 A.M.

FORMAL OPENING OF THE SIXTIETH ANNUAL MEETING, PRESIDENT R. A. SCHATZEL

The session was formally opened by President R. A. Schatzel, who called attention to the very complete program of events that were to follow during the ensuing week. The meeting was then turned over to the session chairman, L. L. Wyman, National Bureau of Standards.

## GENERAL TESTING SESSION

SESSION CHAIRMAN: L. L. WYMAN

***Papers***

Autographic Stress-Strain Recorders—R. R. Bouche and D. R. Tate, presented by Mr. Bouche.[2]

Determination of Strain-Hardening Characteristics by Torsion Testing—D. S. Fields, Jr., and W. A. Backofen, presented by Mr. Fields.

An Evaluation of Geiger Counter X-Ray Techniques for Measuring Stresses in Hardened Steel—Karl E. Beu, presented by title only.

Nondestructive Technique for Macro and Micrographic Surface Examination of Metallic Specimens (English Translation)—A. Jacquet, presented by Chairman L. L. Wyman.

What Can Be Done to Improve Alloy Phase Nomenclature—P. A. Beck, presented by Mr. W. L. Fink.[2a]

Constant Strain-Rate Testing Machine with Instantaneous Speed Change—M. J. Manjoine, E. T. Wessel, and W. H. Pryle, presented by Mr. Manjoine.[2a]

The Significance of Test Results from Small Groups of Specimens—E. H. Schuette, presented by the author.

***Committee E-1 on Methods of Testing:***

Report presented by J. R. Townsend, chairman, and the following actions taken:

*Accepted as Tentative:*

Method for Inspection and Verification of Hydrometers (E 126 - 57 T)

Spec. for Gravity-Convection and Forced-Ventilation Laboratory Ovens (E 127- 57 T)[3]

Test for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use (E 128 - 57 T)

*Accepted as Tentative, Revisions in:*

Methods of Verification of Testing Machines (E 4 - 55 T)

Method of Verification and Classification of Extensometers (E 83 - 52 T)

Spec. for ASTM Hydrometers (E 100 - 55 T), except that the proposal to delete the requirements for specific gravity hydrometer 90H should be omitted.

Method of Bend Testing for Ductility of Metals (E 16 - 39)

Test for Thickness of Solid Electrical Insulation (D 374 - 42)

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[2] Published in the ASTM BULLETIN, No. 228, Feb. (1958).

[2a] Published in the ASTM BULLETIN, No. 226, Dec., 27, and 31 (TP 219), respectively.

[3] The letter ballot in the committee on these specifications resulted in negative votes, and in view of this they are being published as information only rather than as a tentative. See p. 429.

*Adopted as Standard:*

Methods of Verification of Calibration Devices for Verifying Testing Machines (E 74 – 55 T)
Rec. Practice for Operation of Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Artificial Weathering Test (E 42 – 55 T)
Test for Saybolt Furol Viscosity of Asphaltic Materials at High Temperatures (E 102 – 54 T)

*Adopted as Standard, Revisions in:*

Spec. for ASTM Thermometers (E 1 – 56)
Test for Diamond Pyramid Hardness of Metallic Materials (E 92 – 55)

*Accepted for Publication as Information Only:*

Spec. for Distillation Equipment

## SECOND SESSION—SOILS

MONDAY, JUNE 17, 2:00 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT K. B. WOODS

The Use of a Field Vane Apparatus in Sensitive Clay—W. J. Eden and J. J. Hamilton, presented by Mr. Eden.[3a]
The Permeability of Soils and the Concept of a Stationary Boundary-Layer—W. E. Schmid, presented by the author.
Soil Stabilization by Chemical Grouting—R. H. Karol, presented by the author.
Design and Deflection Control of Buried Steel Pipe Supporting Earth Loads and Live Loads—Russell Barnard, presented by the author.

## ROUND-TABLE DISCUSSION ON STATISTICAL ASPECTS OF ASTM STANDARDS

MONDAY, JUNE 17, 2:00 P.M.

*Under auspices of Committee E-11 on Quality Control*
H. F. Dodge, Chairman
O. P. Beckwith, Secretary
*Sponsored by Subcommittee I on ASTM Problems*
W. R. Pabst, Jr., Chairman

This round-table session was arranged in order to provide an opportunity for members of the Society and particularly of the technical committees to discuss their mutual problems in the field of statistical analysis and quality control techniques. To initiate the discussion the following three papers were presented:

Planning Inter-Laboratory Tests—presented by Grant Wernimont.
Selecting the More Sensitive Test Method—presented by John Mandel.
Review of the E-11 Program—presented by Oliver P. Beckwith.

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[3a] Published in *ASTM STP No. 193.*

## THIRD SESSION—COMMITTEE REPORT SESSION

MONDAY, JUNE 17, 4:30 P.M.

SESSION CHAIRMAN: M. A. PINNEY

***Committee C-17 on Asbestos-Cement Products:***

Report presented by W. V. Friedlaender, chairman, and accepted as a report of progress.

***Committee D-13 on Textile Materials:***

Report presented by B. L. Whittier, chairman, and the following actions taken:

*Accepted as Tentative:*

Test for Thermal Transmittance of Textile Fabric and Batting Between Guarded Hot-Plate and Cool Atmosphere (D 1518 - 57 T)
Test for Dimensional Change of Knit Fabrics (D 1470 - 57 T)
Methods of Testing Tufted Pile Floor Coverings (D 1486 - 57 T)
Test for Predicting Differential Dyeing Behavior of Cotton (D 1464 - 57 T)
Test for Shrinkage in Laundering and Dimensional Restorability of Warp Knit Fabrics (D 1487 - 57 T)

*Accepted as Tentative, Revisions in:*

Methods of Quantitative Analysis of Textiles (D 629 - 56 T)
Methods of Testing and Tolerances for Cotton Yarns (D 180 - 54 T)
Methods of Identification of Fibers in Textiles (D 276 - 56 T)
Rec. Practice for Designation of Yarn Construction (D 1244 - 53 T)
Methods of Testing Spun and Filament Yarns Made Wholly or in Part of Man-Made Organic Base Fibers (D 1380 - 56 T), with the following additional change:

*Section 25.*—Replace the revision of this section in the preprinted report with the following revision of Paragraph (*a*):

(*a*) The testing machine for strength or extensibility determinations, or both, may be of the constant-rate-of-traverse (pendulum), constant-rate-of-load, or constant-rate-of-specimen-extension types. Such machines shall conform to the Specifications for Textile Testing Machines (ASTM Designation: D 76)[3] with respect to tolerances, load indication, working range and capacity-verification, and verification of recorded elongation. Clamps with flat-faced jaws shall be used. They may be operated by cams or pneumatically. If, however, (*1*) the specimens slip through the clamps, or (*2*) more than 20 per cent of the specimens break between the jaw faces or closer than $\frac{1}{8}$ in. to the nips of the clamps, then snubbing-type clamps shall be used. The snubbing surfaces shall be fixed and integral with the jaw face. They may be circular, having a diameter of not less than 0.5 in., or semi-involute.

*Section 32* (*a*).—Change to read as set forth above for Section 25(*a*).

Test for Average Fiber Diameter of Wool Tops by the Porous Plug Tester (D 1282 - 53 T)
Spec. and Method of Test for Fineness of Mohair Tops (D 1381 - 55 T)
Def. of Terms Relating to Textile Materials (D 123 - 55)
Methods of Testing and Tolerances for Cotton Sewing Threads (D 204 - 56)
Methods of Testing Rayon and Acetate Staple (D 540 - 44)

*Adopted as Standard:*

Test for Wool Content of Raw Wool (Laboratory Scale) (D 584 - 54 T)
Test for Alkali-Solubility of Wool (D 1283 - 53 T)
Test for Strength of Cotton Fibers (Flat Bundle Method) (D 1445- 53 T)
Test for Maturity of Cotton Fibers (Polarized-Light Method) (D 1450 - 55 T)
Test for Magnetic Rating of Asbestos Used for Electrical Purposes (D 1118 - 50 T)

*Adopted as Standard, Revisions in:*

Methods of Testing Felt (D 461 - 53)
Test for Resistance of Textile Materials to Microorganisms (D 684 - 54)
Test for Evaluating Treated Textiles for Permanence of Resistance to Microorganisms (D 862 - 54)
Test for Yarn Number of Yarn from Fabrics (D 1059 - 56)
Spec. and Methods of Test for Asbestos Lap (D 1061 - 54)
Methods of Testing Bonded Fabrics (D 1117 - 53)

*Tentative Withdrawn:*

Test for Absorbency Time and Absorptive Capacity of Nonwoven Fabrics (D 1378 - 55 T)

*Tentative Revision Withdrawn:*

Methods of Testing Rayon and Acetate Staple (D 540 - 44)

*Standards Withdrawn:*

Test for Shrinkage in Laundering of Knit Cotton Fabrics (D 1231 - 54)
Test for Shrinkage in Laundering of Knit Rayon Fabrics (D 1232 - 54)

*Accepted for Publication as Information Only:*

Test for Length and Length Distribution of Cotton Fibers by the Short Array Method Test for Estimating Maturity and Weight Per Unit Length of Cotton Fibers by the Causticaire Method

***Committee D-22 on Methods of Atmospheric Sampling and Analysis:***

Report presented in the absence of the chairman by R. J. Painter, and the following actions taken:

*Adopted as Standard:*

Method for Determining Odor Concentration in Air and Gases (Dilution Method) (D 1391 - 56 T)
Rec. Practice for Planning the Sampling of the Atmosphere for Analysis (D 1357 - 55 T)

***Committee D-23 on Cellulose and Cellulose Derivatives:***

Report presented in the absence of the chairman by R. J. Painter, and the following action taken:

*Adopted as Standard:*

Method of Testing Cellulose Acetate Butyrate (D 817 - 55 T) (Jointly with Committee D-20)

***Committee E-12 on Appearance:***

Report presented in the absence of the chairman by G. W. Ingle, vice-chairman, and accepted as a report of progress.

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## FOURTH SESSION—STEEL

MONDAY, JUNE 17, 8:00 P.M.

SESSION CHAIRMAN: C. L. CLARK

The Effect of Phosphorus on the Susceptibility to Temper Embrittlement of Cast Cr-Mo-V Steel—J. Chaberek and R. S. Zeno, presented by Mr. Zeno.
The Static Properties of Several High-Strength Steels—E. P. Klier, B. B. Muvdi and G. Sachs, presented by Mr. Klier.
Behavior of Open Hearth and Bessemer Seamless Steel Pipe—A. B. Wilder and W. P. Benter, presented by Mr. Benter.
A Laminated Specimen for Charpy Impact Testing of Sheet Metal—S. V. Arnold, presented by the author.

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## FIFTH SESSION—FATIGUE

MONDAY, JUNE 17, 8:00 P.M.

SESSION CHAIRMAN: F. M. HOWELL

Cyclic Strain Fatigue Studies on AISI Type 347 Stainless Steel—E. E. Baldwin, G. Sokol, and L. G. Coffin, Jr., presented by Mr. Coffin.
The Fatigue Properties of Decarburized Steel—G. T. Horne and H. A. Lipsitt, presented by Mr. Lipsitt.
Cracking of Notch Fatigue Specimens—M. S. Hunter and W. G. Fricke, presented by Mr. Fricke.
Effect of Grinding Conditions and Resultant Residual Stresses on the Fatigue Strength of Hardened Steel—L. P. Tarasov, W. S. Hyler, and H. R. Letner, presented by Mr. Tarasov.
Fretting Fatigue Strength of Titanium Alloy RC 130 B—H. W. Liu, H. T. Corten, and G. M. Sinclair, presented by Mr. Liu.

## SIXTH SESSION—CONCRETE

MONDAY, JUNE 17, 8:00 P.M.

SESSION CHAIRMAN: L. E. GREGG

The Freeze-Thaw Resistance of Concrete as Affected by the Method of Test—H. L. Flack, presented by the author.

Instrumentation and Methods for Pairwise Impulse Testing of Reinforced Concrete Beams—F. T. Mavis and M. J. Greaves, presented by Mr. Greaves.[4]

Performance Tests of Concrete Truck Mixers—Albert G. Timms, presented by the author.

Cement-Aggregate Reaction in Concrete of a Canadian Bridge—E. G. Swenson, presented by the author.

A Canadian Reactive Aggregate Undetected by ASTM Tests—E. G. Swenson, presented by the author.[4]

Designing Progressive Specifications for Quality Control—E. A. Abdun-Nur and L. H. Tuthill, presented by Mr. Tuthill. (For Presentation Only)

*(Continued in the Sixteenth Session)*

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[4] Published in the ASTM BULLETIN, No. 226, Dec., 43 (TP 231), and 48 (TP 236), respectively.

## SEVENTH SESSION—SYMPOSIUM ON DETERMINATION OF GASES IN METALS[5]

TUESDAY, JUNE 18, 9:30 A.M.

SESSION CHAIRMAN: F. M. FOWLER

Introduction by F. M. Fowler.

Two Apparati for the Determination of Gases in Metals—D. L. Guernsey and R. H. Franklin, presented by Mr. Guernsey.

Application of Vacuum Fusion to Gas-Metal Studies—W. G. Guldner and A. L. Beach, presented by Mr. Guldner.

Oxygen Determinations Using a Platinum Bath and Capillary Trap—W. G. Smiley, presented by the author.

Bromination-Carbon Reduction Method for the Determination of Oxygen in Metals—Maurice Codell and George Norwitz, presented by Mr. Codell.

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[5] Issued as separate publication *ASTM STP No. 222.*

## EIGHTH SESSION—SYMPOSIUM ON LARGE FATIGUE TESTING MACHINES AND THEIR RESULTS[6]

TUESDAY, JUNE 18, 9:30 A.M.

SESSION CO-CHAIRMEN: D. G. RICHARDS
O. J. HORGER

Introduction by J. M. Lessells.

A Unique Machine for Large Scale Fatigue Testing—H. V. Cordiano, presented by the author.

Torsional Fatigue Testing of Axle Shafts—Edwin J. Eckert, presented by the author.

Fatigue Testing of Airframe Structural Components—H. W. Foster, presented by the author.

*(Symposium Continued in the Tenth Session)*

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[6] Issued as separate publication *ASTM STP No. 216.*

## NINTH SESSION—LUNCHEON, REPORT OF BOARD OF DIRECTORS, RECOGNITION OF 50-YEAR MEMBERS, PRESIDENT'S ADDRESS, INTRODUCTION OF NEW OFFICERS

TUESDAY, JUNE 18, 12:00 NOON

CHAIRMAN: PAST-PRESIDENT N. L. MOCHEL

About 225 were present at this luncheon session, including a number of ladies and visitors.

### *Report of Board of Directors:*

R. J. Painter, Executive Secretary, presented a brief résumé of the Report of the Board of Directors.

### *Recognition of 50-Year Members:*

Chairman Mochel read the names of the nine members, both individual and company, who had been continuously affiliated with the Society for 50 years, and certificates to that effect were presented. Dr. John Fairfield Thompson, Chairman of the Board of the International Nickel Co. of Canada, Ltd., made a brief, fitting response on behalf of the 50-Year Members. His remarks are published in the July, 1957, ASTM BULLETIN.

### *Presidential Address:*

The Annual President's Address was presented by R. A. Schatzel, on the subject of "Our Expanding Technology and ASTM."

### *Election of Officers:*

Results of the letter ballot on election of new officers were announced by F. H. Pennell on behalf of the Teller's Committee consisting of Mr. Pennell, Chairman, and Walter Clavan. The results were as follows:

For President, to serve for one year:
  R. T. Kropf, 2099 votes.
For Vice-President, to serve for two years:
  F. L. LaQue, 2105 votes.
For Directors, to serve for three years:
  C. L. Clark, 2093 votes.
  A. E. Juve, 2086 votes.
  J. H. Koenig, 2084 votes.
  R. E. Peterson, 2087 votes.
  R. W. Seniff, 2080 votes.

The newly-elected members of the Board of Directors who were present were introduced, as were President-elect Kropf, Vice-President-elect LaQue, and Vice-President Woods (continuing for another year). The latter three responded briefly. Photographs and short biographical sketches of the new officers appear in the July, 1957, ASTM BULLETIN.

### *Special Presentations:*

At the close of the session, the Chairman presented the Past-President's pin to retiring President R. A. Schatzel. Mention was made that this meeting brings to a close for L. C. Beard, Jr., nine years on the Board of Directors, as Director, Vice-President, President, and Past-President. Recognition was also given to E. J. Albert, J. M. Campbell, P. V. Garin, J. H. Jenkins and D. E. Parsons as retiring Directors.

## TENTH SESSION—SYMPOSIUM ON LARGE FATIGUE TESTING MACHINES AND THEIR RESULTS[6]

*(Symposium Continued from Eighth Session)*

TUESDAY, JUNE 18, 2:30 P.M.

SESSION CO-CHAIRMEN: J. F. MILLAN
H. J. GROVER

Fatigue Performance of Marine Shafting Laboratory and Service Tests—R. W. Bunyan, presented by the author.

Fretting Corrosion of Large Shafts as Influenced by Surface Treatments—O. J. Horger and H. R. Neifert, presented by Mr. Neifert.

Fatigue Tests of Large Alloy Steel Shafts—F. C. Eaton, presented by the author.

A Quarter Century of Propulsion Shafting Design Practice and Operating Experience in the U. S. Navy—R. Michel, presented by the author.

## ELEVENTH SESSION—SYMPOSIUM ON SPECTROCHEMICAL ANALYSIS FOR TRACE ELEMENTS[7]

TUESDAY, JUNE 18, 2:30 P.M.

SESSION CHAIRMAN: D. L. TIMMA

Emission Spectrometric Determination of Oxygen in Metals—V. A. Fassel, W. A. Gordon and R. W. Tabeling, presented by Mr. Fassel.

Spectrographic Determination of Trace Elements in Metals—J. A. Norris, presented by the author.

Trace Analysis by Means of the Graphite Spark—James M. Morris and Francis X. Pink, presented by Mr. Morris.

*(Symposium Continued in the Fourteenth Session)*

## SESSION ON ELECTRON METALLOGRAPHY

TUESDAY, JUNE 18, 2:30 P.M.

SESSION CHAIRMAN: S. T. ROSS

Subcommittee XI on Electron Metallography of Metals of Committee E-4 on Metallography held a technical session on electron metallography, and the following papers were presented:

The First Progress Report of the Non-Ferrous Task Group on the Electron Microstructure of an 8% Manganese Titanium-Base Alloy.

Procedure for the Preparation of Shadowed Plastic Replicas—S. T. Ross, D. M. Teague, and G. E. Pelissier.

The Two-Stage Carbon Replica for Electron Metallography—R. L. Scott.

Observations on the Dislocation Structure of Sub-Boundaries in Iron—F. W. Boswell.

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[7] Issued as separate publication *ASTM STP No. 221.*

## TWELFTH SESSION—COMMITTEE REPORT SESSION

TUESDAY, JUNE 18, 4:30 P.M.

SESSION CHAIRMAN: R. W. SENIFF

### *Committee A-9 on Ferro-Alloys:*

Report presented by S. W. Poole, chairman, and the following action taken:

*Accepted as Tentative, Revisions in:* .

Spec. For Ferromanganese (A 99 - 53 T)

### *Committee B-7 on Light Metals and Alloys, Cast and Wrought:*

Report presented by I. V. Williams, chairman, and the following actions taken:

*Accepted as Tentative:*

Spec. for Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 - 57 T), revised as follows:

*Fig. 1.*—Change the figure title to read, "Double shear jig for rivet and cold heading wire." Change the hole diameter for $\frac{3}{8}$ in. nominal rivet size from "0.3475" to read "0.3745."

Spec. for Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors) (B 317 - 57 T), revised as follows:

*Section 2(d).*—In the last line delete the word "tees."

*Table XVII.*—Change heading of second column to read, "Permissible variation, deg." Change the wording in the first column from "20 and under" to read "all." Change the tolerance value in the second column from "$\frac{1}{4}$" to read "1."

Spec. for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 - 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Aluminum-Base Alloy Sand Castings (B 26 - 56 T)

Spec. for Magnesium-Base Alloy Sand Castings (B 80 - 56 T)

Spec. for Magnesium-Base Alloy Sheet (B 90 - 56 T)

Spec. for Magnesium-Base Alloy Forgings (B 91 - 56 T)

Spec. for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 - 56 T)

Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 56 T)

Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 56 T)

Spec. for Magnesium-Base Alloy Permanent Mold Castings (B 199 - 56 T)

Spec. for Aluminum-Alloy Sheet and Plate (B 209 - 56 T), with the following additional change:

*Section 7.*—Change to read as follows:

7. Sheet and plate shall be supplied in the alloy, temper, and size specified in the contract or order and shall conform to the requirements as to tensile properties prescribed in Table II. Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 990A, 996A, G1A, G1B, GM40A, GM41A, GM51A, GR20A, GR40A, M1A, clad M1A, GM11A, and clad MG11A, yield strength is not important. For these alloys, yield strength need not be determined unless specifically indicated in the contract or purchase order.

Spec. for Aluminum-Alloy Drawn Seamless Tubes (B 210 - 56 T), with the following additional change:

*Table II.*—Change Footnote *d* to read, "Annealed (O) temper tube shall upon heat treatment be capable of developing the mechanical properties applicable to T3 temper tube except that the minimum yield strength shall be 40,000 psi."

Spec. for Aluminum-Alloy Bars, Rods, and Wire (B 211 - 56 T)

Spec. for Magnesium-Base Alloy Extruded Tubes (B 217 - 56 T)

Spec. for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 56 T), with the following additional changes:

*New Table VII.*—Change the heading of the second column from "minimum" to read "Up through 5 ft." Change the heading of the third column from "maximum" to read "Over 5 ft."

Spec. for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 56 T)

Spec. for Aluminum-Alloy Extruded Tubes (B 235 - 56 T)

Spec. for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 - 56 T)

Spec. for Aluminum-Alloy Pipe (B 241 - 56 T)
Spec. for Aluminum-Alloy Die Forgings (B 247 - 55 T)
Spec. for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Application (B 307 - 56 T)
Spec. for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 - 56 T), with the following additional changes:

*Table I.*—Add chemical requirements for alloy GM51A as indicated for Table I of Specification B 221, and add chemical requirements for alloy GS11C indicating the same composition limits as now shown for alloy GS11A except chromium limits are to be 0.04–0.14 per cent.

*Appendix.*—Add to the Table of Cross Index of ASTM and AA designations, ASTM alloys GM51A and GS11C with corresponding AA designations 5456 and 6062, respectively.

Spec. for Aluminum-Alloy Round Welded Tubes (B 313 - 56 T)

*Adopted as Standard, Revisions in:*

Spec. for Aluminum for Use in Iron and Steel Manufacture (B 37 - 49)
Spec. for Magnesium Ingot and Stick for Remelting (B 92 - 56)
Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 56)

*Tentatives Withdrawn:*

Spec. for Aluminum-Alloy Sheet and Plate for Pressure Vessel Applications (B 178 - 56 T)
Spec. for Aluminum-Alloy Bars, Rods, and Shapes for Pressure Vessel Applications (B 273 - 56 T)
Spec. for Aluminum-Alloy Pipe and Tube for Pressure Vessel Applications (B 274 - 56 T)

### *Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements:*

Report presented in the absence of the chairman by S. F. Etris, and the following action taken:

*Adopted as Standard:*

Test for Ignition Loss and Active Calcium Oxide in Magnesium Oxide for Use in Magnesium Oxychloride Cements (C 247 - 54 T)

### *Committee D-8 on Bituminous Waterproofing and Roofing Materials:*

Report presented by H. R. Snoke, chairman, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 - 54 T)
Spec. for Woven Burlap Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1327 - 54 T)
Spec. for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-up Roofs (D 250 - 56)
Spec. for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-up Roofs (D 655 - 47)
Spec. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 - 47)
Spec. for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 - 44)

### *Joint Committee on Leather:*

Report presented by J. R. Kanagy, chairman, and the following actions taken:

*Accepted as Tentative:*

Test for Area of Leather (D 1515 - 57 T), with the following change:

*Section 3.* Add as the second sentence, "It shall be conditioned for 48 hr in an atmosphere maintained at 73.5 ± 2 F (23 ± 1 C) and 50 ± 4 per cent RH and tested under these conditions.

Test for Width of Leather (D 1516 - 57 T), with Section 3 revised as set forth above for D 1515.
Def. of Terms Relating to Leather (D 1517 - 57 T)

The committee withdrew from the report as preprinted the recommendations for the publication as tentative of the Method of Conditioning Leather and Leather Products for Testing, Method of Test for Corrosion Produced by Leather in Contact with Metal, and Method of Test for Oil or Air Permeability of Leather Cup Packings.

## THIRTEENTH SESSION—GILLETT MEMORIAL LECTURE[8]

TUESDAY, JUNE 18, 5:00 P.M.

SESSION CHAIRMAN: PRESIDENT R. A. SCHATZEL

***Sixth Gillett Memorial Lecture:***

President Schatzel called upon Mr. H. C. Cross of Battelle Memorial Inst. who remarked upon the establishment of the H. W. Gillett Memorial Lecture. This lecture is sponsored jointly by the ASTM and the Battelle Memorial Inst. to perpetuate the memory of Horace W. Gillett, one of the America's leading technologists, the first Director of the Institute, and for many years a very active worker in the Society, through the presentation of a lecture on a subject pertaining to the development, testing, evaluation, and application of metals.

Mr. Cross introduced Alvin J. Herzig, President, Climax Molybdenum Co. of Michigan, who presented the sixth H. W. Gillett Memorial Lecture on the subject: "A Perspective of Molybdenum-Base Alloys." The lecturer gave a perspective of the broad subject not only by reciting some of the properties which have already been achieved in molybnum-base alloys but also by speculation in some of the areas where complex, technical problems still remain. One of the several furnaces now being employed to obtain aggregates of molybdenum by melting rather than by the powder metallurgy processes was described in some detail. Also there was a short motion picture of the action at the molten metal surface during the production of a 1000-lb ingot of molybdenum.

President Schatzel expressed appreciation to Mr. Herzig for his outstanding lecture and presented to him on behalf of the Society the H. W. Gillett Memorial Lecture Certificate.

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[8] Issued as a separate publication.

## FOURTEENTH SESSION—SYMPOSIUM ON SPECTROCHEMICAL ANALYSIS FOR TRACE ELEMENTS[7]

(*Symposium Continued from Eleventh Session*)

TUESDAY, JUNE 18, 8:00 P.M.

SESSION CHAIRMAN: D. L. TIMMA

Medical and Biological Materials—Bert Vallee, presented by the author.

Plants and Soils—W. G. Schrenk, presented by the author.

Geological Materials—K. J. Murata, presented by the author.

## FIFTEENTH SESSION—NON-FERROUS METALS

TUESDAY, JUNE 18, 8:00 P.M.

SESSION CHAIRMAN: F. L. LAQUE

Ultrasonic Testing of Aluminum Alloys—W. L. Fink, presented by the author.[9]

Electrical Resistivity and Thermoelectric Potential of Rhenium Metal—G. B. Gaines and C. T. Sims, presented by Mr. Sims.

Mechanical Behavior of Cemented Carbides—J. D. Lubahn and R. P. Felgar, presented by Mr. Lubahn.

The Directionality of Beryllium Copper Strip as Affected by Cold Rolling and Heat Treating—J. T. Richards, presented by the author.

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[9] Published in the ASTM BULLETIN, No. 226, Dec., 36 (TP 224).

## SIXTEENTH SESSION—CONCRETE

(*Continued from Sixth Session*)

TUESDAY, JUNE 18, 8:00 P.M.

SESSION CHAIRMAN: R. R. LITEHISER

Insulating Concrete and Compressive Strength—Morton Sherman, presented by the author.

Studies of Flexural Strength of Concrete. Part 3. Effects of Variations in Testing Procedures—Stanton Walker and D. L. Bloem, presented by Mr. Bloem.

Measuring the Rate of Hardening of Concrete by Bond Pullout Pins—T. M. Kelly and D. E. Bryant, presented by Mr. Kelly.

Wear Tests on Concrete Using the German Standard Method of Test and Machine—James L. Sawyer, presented by the author.

## SEVENTEENTH SESSION—HIGH-TEMPERATURE TESTING

WEDNESDAY, JUNE 19, 9:30 P.M.

SESSION CHAIRMAN: J. D. LUBAHN

***Joint Committee on Effect of Temperature on the Properties of Metals:***

Report presented by V. T. Malcolm, chairman, and accepted as a report of progress.

***Papers:***

Embrittling Tendencies of Austenitic Superheater Materials at Elevated Temperatures—J. H. Hoke, F. Eberle, and R. D. Wylie, presented by Mr. Hoke.

Influence of Notch Preparation and Eccentricity of Loading on the Notch Rupture Life—M. H. Jones, J. L. Shannon, and W. F. Brown, Jr., presented by Mr. Brown.

Constant Stress, Creep-Rupture Tests of a Killed Carbon Steel—Pryor N. Randall, presented by the author.

Microhardness Testing at High Temperatures—J. W. Westbrook, presented by the author.

Thermal Shock Resistance of High-Temperature Alloys—F. L. Muscatell, E. E. Reynolds, W. W. Dyrkacz and J. H. Dalheim, presented by Mr. Reynolds.

(*Continued in the Twentieth Session*)

## EIGHTEENTH SESSION—PAPER ON BITUMINOUS MIXTURES AND COMMITTEE—REPORT SESSION

WEDNESDAY, JUNE 19, 11:15 A.M.

CHAIRMAN: D. E. PARSONS

***Paper:***

A Cooperative Investigation of the Method of Test for Compressive Strength of Bituminous Mixtures—C. E. Proudley and H. Fred Waller, Jr., presented by Mr. Waller.

***Committee C-3 on Chemical-Resistant Mortars:***

Report presented in the absence of the chairman by J. Roy Allen, vice-chairman, and the following actions taken:

*Accepted as Tentative:*

Spec. for Resin-Type Chemical-Resistant Mortars (C 395 - 57 T), with the following change:

*Section 7.*—Delete Paragraphs (*e*), (*f*) and (*g*) and the note to this section.

Test for Compressive Strength of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 396 - 57 T)

Rec. Practice for Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 397 - 57 T)

Rec. Practice for Use of Hydraulic Cement Mortars (C 398 - 57 T)

Rec. Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 - 57 T)

*Adopted as Standard, Revisions in:*

Test for Bond Strength of Chemical-Resistant Mortars (C 321 - 56)

### Committee C 7 on Lime:

Report presented in the absence of the chairman by E. E. Eakins, vice-chairman, and the following actions taken:

*Accepted as Tentative:*

Method of Testing Quicklime and Hydrated Lime for Neutralization of Waste Acid (C 400 - 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Inorganic Aggregates for Use in Interior Plaster (C 35 - 54 T) (Jointly with Committee C-11)

*Adopted as Standard, Revisions in:*

Spec. for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 - 42)

Methods of Sampling, Inspection, Packing, and Marking of Quicklime and Lime Products (C 50 - 27)

Methods of Physical Testing of Quicklime and Hydrated Lime (C 110 - 49)

*Standard Withdrawn:*

Spec. for Hydrated Lime for Varnish Manufacture (C 47 - 27)

### Committee C-9 on Concrete and Concrete Aggregates:

Report presented by W. H. Price, chairman, and the following actions taken:

*Accepted as Tentative:*

Spec. for Raw or Calcined Natural Pozzolans for Use as an Admixture in Portland Cement Concrete (C 402 - 57 T)

Test for Measuring the Rate of Hardening of Mortars Wet-Sieved from Concrete Mixtures by Proctor Penetration Resistance Needles (C 403 - 57 T)

*Accepted as Tentative, Revisions in:*

Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland Cement Concrete (C 311 - 54 T)

Spec. for Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350 - 54 T)

Test for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293 - 54 T)

Test for Lightweight Pieces in Aggregate (C 123 - 53 T)

Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 - 54 T)

Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 - 56 T)

Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 - 56 T)

Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 - 56 T)

Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 - 56 T)

Test for Scratch Hardness of Coarse Aggregate Particles (C 235 - 54) (Jointly with Committee D-4)

Test for Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel (C 234 - 54)

*Adopted as Standard:*

Spec. for Concrete Aggregates (C 33 - 56 T)

*Adopted as Standard, Revisions in:*

Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 - 55)

Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 - 49)

Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 - 49)

Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 55)
Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 42)

### *Committee C-13 on Concrete Pipe:*

Report presented by R. R. Litehiser, chairman, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Spec. for Reinforced Concrete Low-Head Pressure Pipe (C 361 – 55 T), with the following additional change:

*Section 30(a).*—In line 14, following "36 in.," add "inclusive, or more than 3 per cent for pipe having internal diameters longer than 36 in."

*Adopted as Standard, Revisions in:*

Spec. for Concrete Sewer Pipe (C 14 – 56)

### *Committee C-18 on Natural Building Stones:*

Report presented in the absence of the chairman by L. C. Gilbert, and the following actions taken:

*Accepted as Tentative:*

Spec. for Roofing Slate (C 406 – 57 T), with the following change:

*Section 4 (a).*—Change the third sentence to read, "Broken corners on the exposed ends of shingles may be considered cause for rejection when either the base or leg of the right triangular piece broken off is greater than $1\frac{1}{2}$ in."

*Accepted for Publication as Information Only:*

Proposed Spec. for Structural Granite

### *Committee C-19 on Structural Sandwich Constructions:*

Report presented in the absence of the chairman by L. C. Gilbert, and the following actions taken:

*Accepted as Tentative:*

Test for Flatwise Flexure Strength of Sandwich Construction (C 393 – 57 T)
Test for Shear Fatigue of Sandwich Core Materials (C 394 – 57 T)

*Adopted as Standard:*

Test for Delamination Strength of Honeycomb Type Core Material (C 363 – 55 T)
Test for Edgewise Compressive Strength of Flat Sandwich Construction (C 364 – 55 T)
Test for Flatwise Compressive Strength of Sandwich Cores (C 365 – 55 T)
Test for Measurement of Thickness of Sandwich Cores (C 366 – 55 T)

### *Committee C-20 on Acoustical Materials:*

Report presented in the absence of the chairman by L. C. Gilbert, and the following action taken:

*Adopted as Standard:*

Test for Strength Properties of Prefabricated Architectural Acoustical Materials (C 367 – 55 T)

### *Committee E-6 on Methods of Testing Building Constructions:*

Report presented in the absence of the chairman by L. C. Gilbert, and accepted as a report of progress.

## NINETEENTH SESSION—AWARDS LUNCHEON—AWARDS OF MERIT, DUDLEY MEDAL, TEMPLIN, TOUR, RICHART, HECHT AND THOMPSON AWARDS, RECOGNITION OF 40-YEAR MEMBERS

WEDNESDAY, JUNE 19, 12:00 NOON

CHAIRMAN: VICE-PRESIDENT R. T. KROPF

About 280 were present at this Awards Luncheon, including a number of ladies and visitors.

### *Awards of Merit:*

The Chairman of the 1957 Award of Merit Committee, M. A. Swayze, read brief citations and presented the following to President Schatzel, who conferred on them, on behalf of the Board of Directors, the Certificate of Award of Merit:

*John W. Bolton
Donald L. Colwell
A. G. H. Dietz
*Theodore P. Dresser, Jr.
Paul V. Faragher
Edgar W. Fasig
William A. Kennedy (posthumously)
Wilmer H. Koch
R. Earl Penrod
Frank W. Reinhart
Francis G. Tatnall
Walter C. Voss

* Presented in absentia.

### *Charles B. Dudley Medal Award:*

The Twenty-seventh Charles B. Dudley Medal Award was made to R. U. Blaser, Assistant Superintendent, Materials Division, Babcock & Wilcox Co., Research Center, Alliance, Ohio, and J. J. Owens, formerly with Babcock & Wilcox Co., now with Thompson Products, for their paper on "Special Corrosion Study of Carbon and Low-Alloy Steels," published in the 1955 Proceedings.

### *Richard L. Templin Award:*

The Eleventh Richard L. Templin Award was made to W. Ramberg and L. K. Irwin, Chief, Division of Mechanics and Mechanical Engineer, respectively, Engineering Mechanics Section, National Bureau of Standards, Washington, D. C., for their paper on "Longitudinal Impact Tests of Long Bars with a Slingshot Machine," published in the 1955 Proceedings.

### *Sam Tour Award:*

The Sixth Sam Tour Award was made to K. G. Compton and A. Mendizza, Mechanical Staff, Bell Telephone Laboratories, Inc., for their paper on "Galvanic Couple Corrosion Studies by Means of the Threaded Bolt and Wire Test," published in the 1955 Proceedings.

### *Frank E. Richart Award:*

The Second Frank E. Richart Award was made to Stanton Walker, Director of Engineering, National Sand and Gravel Association, Washington, D. C., who has made notable contributions to the work of the Society in the field of concrete and concrete aggregates.

### *Max Hecht Award:*

The Fourth Max Hecht Award was made to R. C. Adams, Superintendent, Chemical Engineering Laboratory, U. S. Naval Engineering Experiment Station, Annapolis, Md., who has performed outstanding work in Committee D-19 and in the field of industrial water.

### *Sanford E. Thompson Award:*

The Thirteenth Sanford E. Thompson Award was made to T. C. Powers, Manager, Basic Research Section, Portland Cement Association, Chicago, Ill., for his outstanding work in the field of concrete and concrete aggregates.

### *Recognition of 40-Year Members:*

Chairman Kropf read the names of the 31 members, both individual and company, who had been continuously affiliated with the Society for 40 years, and certificates to that effect were presented.

## TWENTIETH SESSION—HIGH-TEMPERATURE TESTING

(*Continued from the Seventeenth Session*)

WEDNESDAY, JUNE 19, 2:30 P.M.

SESSION CHAIRMAN: H. C. CROSS

An Experimental Study of Compressive-Creep Behavior at Elevated Temperatures—R. L. Carlson and E. G. Bodine, presented by Mr. Carlson.

Tensile and Creep Properties of Tungsten at Elevated Temperatures—J. W. Pugh, presented by the author.

Dynamic Elastic Modulus Values at Elevated Temperatures for Nickel Base, Aluminum Base and Metal-Metal Oxide Alloys—Robert W. Wilde, presented by the author.

Effect of Composition on the Elevated Temperature Properties of Ductile Iron—R. D. Schelleng and J. T. Eash, presented by Mr. Schelling.

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## DISCUSSION ON NON-COMMERCIAL MICROSCOPIC ACCESSORIES

WEDNESDAY, JUNE 19, 2:30 P.M.

*Sponsored by Subcommittee 28 on Microscopy of Committee E-1 on Methods of Testing*

F. G. Foster, Chairman

A. F. Kirkpatrick, Secretary

M. C. Miller, Chairman of Discussion Session

All microscopists have made some accessories for the optical or electron microscopes, or have developed techniques for preparation and examination of samples; however, they have had little opportunity for the general exchange of information. In accordance with the purposes of Subcommittee 28, on Microscopy, of Committee E-1 on Methods of Testing, to develop standards and methods applicable in industrial microscopy and to collect and distribute microscopical information, this session on accessories was organized.

To stimulate discussion from the floor, several brief presentations were scheduled.

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## TWENTY-FIRST SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 19, 4:00 P.M.

SESSION CHAIRMAN: G. H. HARNDEN

***Committee A-1 on Steel:***

Report presented by W. F. Collins, chairman, and the following actions taken:

*Accepted as Tentative:*

Spec. for High Strength Structural Alloy Rivet Steel (A 406 - 57 T), revised by the deletion of Section 5(*b*).

Spec. for Upholstery Spring Wire for Coiled Type Springs (A 407 - 57 T)

Spec. for Special Large Size Deformed Billet Steel Concrete Reinforcement Bars (A 408 - 57 T), with the following change:

*Section 12*(*d*).—Change to read, "To indicate adequately conformity to the dimensional requirements of Section 7, measurements shall be made at selected points on one bar from each 25 tons or fraction thereof of the lot."[5]

Spec. for Welded Large Outside Diameter Light Wall Austenitic Chromium-Nickel Alloy-Steel Pipe for Corrosive or High-Temperature Service (A 409 - 57 T), with the following change:

*Table I*.—Change the maximum phosphorus content for all grades from "0.030" to read "0.040."

Spec. for Chromium-Copper-Nickel-Aluminum

Alloy-Steel Plates for Pressure Vessels (A 410 - 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Billet-Steel Bars for Concrete Reinforcement (A 15 - 56 T)
Spec. for Rail-Steel Bars for Concrete Reinforcement (A 16 - 54 T)
Spec. for Carbon-Steel Axles for Cars and Tenders (A 21 - 55 T)
Spec. for Welded and Seamless Steel Pipe (A 53 - 55 T)
Spec. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 - 55 T)
Spec. for Electric-Resistance-Welded Steel Pipe (A 135 - 55 T)
Spec. for Axle-Steel Bars for Concrete Reinforcement (A 160 - 54 T)
Spec. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 - 55 T)
Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 - 56 T)
Spec. for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 55 T)
Spec. for Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 - 54 T)
Spec. for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 212 - 54a T)
Spec. for Seamless Alloy-Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 - 56 T)
Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 56 T)
Spec. for Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 - 56 T)
Spec. for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 - 56 T)
Spec. for Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 285 - 54 T)
Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 56 T) (Jointly with Committee A-10)
Spec. for Alloy-Steel Seamless Drum Forgings (A 336 - 55 T)
Spec. for Heavy-Walled Carbon and Low Alloy Steel Castings for Steam Turbines (A 356 - 56 T)
Spec. for Untreated Carbon-Steel Axles for Export and for General Industrial Use (A 383 - 55 T)
Spec. for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 387 - 56 T)
Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts Specially Heat-Treated for High-Temperature Service (A 404 - 56 T)
Spec. for Hard-Drawn Steel Spring Wire (A 227 - 47)

*Adopted as Standard:*

Spec. for High-Strength Structural Rivet Steel (A 195 - 52 T)

*Adopted as Standard, Revisions in:*

Spec. for Wrought Steel Wheels for Electric Railway Service (A 25 - 48)
Spec. for Steel Tires (A 26 - 39)
Spec. for Mild-to-Medium-Strength Carbon-Steel Castings for General Applications (A 27 - 55)
Spec. for Multiple-Wear Wrought Steel Wheels (A 57 - 54)
Spec. for High-Strength Steel Castings for Structural Purposes (A 148 - 55)
Spec. for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 - 56)
Spec. for Carbon Steel Forgings for Railway Use (A 236 - 55)
Spec. for Carbon Steel Seamless Drum Forgings (A 266 - 55)
Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 55) (Jointly with Committee A-10)
Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 56) (Jointly with Committee A-10)
Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 55) (Jointly with Committee A-10)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 56) (Jointly with Committee A-10)
Spec. for Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 - 56)
Spec. for Heat-Treated Steel Tires (A 329 - 54)
Spec. for Forged or Rolled Carbon and Alloy-Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 - 55)

Committee A-1 withdrew from the report as preprinted the recommendation for the revision of Tentative Spec. for Carbon and Alloy-Steel Forgings for Turbine Rotors and Shafts (A 293 - 55 T).

### *Committee B-1 on Wires for Electrical Conductors:*

Report presented in the absence of the chairman by C. E. Topping, and the following actions taken:

*Accepted as Tentative:*

Spec. for Aluminum Wire for Communication Cable (B 314 – 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Concentric-Lay-Stranded Aluminum Conductors, Steel Reinforced (ACSR) (B 232 – 56 T). The paragraphs of Section 5 to be revised are paragraphs (*g*) and (*h*) rather than paragraphs (*f*) and (*g*) as indicated in the preprinted report; the paragraph to be deleted is paragraph (*i*) rather than paragraph (*h*), and the paragraph to be relettered is paragraph (*j*).

Spec. for Soft or Annealed Coated Copper Conductors for Use in Hookup Wire for Electronic Equipment (B 286 – 54 T)

*Adopted as Standard:*

Spec. for Standard Nominal Diameters and Cross-Sectional Areas of AWG Sizes of Solid Round Wires Used as Electrical Conductors (B 258 – 51 T)

*Adopted as Standard, Revisions in:*

Spec. for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (B 48 – 55), with Note 12, referred to at the end of new Section 9, reading as follows:

NOTE 12.—LSE test results are affected by small amounts of cold working. The specified 1 per cent minimum LSE value applies only to bare wire before further processing.

Test for Resistivity of Electrical Conductor Materials (B 193 – 49)

Spec. for Hard-Drawn Copper Covered Steel Wire (B 227 – 52)

Spec. for Concentric-Lay-Stranded Aluminum Conductors, Hard-Drawn and Three-Quarter Hard-Drawn (B 231 – 56)

### *Committee B-2 on Non-Ferrous Metals and Alloys:*

Report presented by Bruce W. Gonser, chairman, and accepted as a report of progress.

### *Committee B-3 on Corrosion of Non-Ferrous Metals:*

Report presented in the absence of the chairman by A. W. Tracy, secretary, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Method for Salt Spray (Fog) Testing (B 117 – 54 T)

Method for Acetic Acid-Salt Spray (Fog) Testing (B 287 – 54 T)

### *Committee B-5 on Copper and Copper Alloys, Cast and Wrought:*

Report presented by G. H. Harnden, chairman, and the following actions taken:

*Accepted as Tentative:*

Spec. for Copper Silicon Alloy Seamless Pipe and Tube (B 315 – 57 T)

*Adopted as Standard:*

Spec. for Copper Rod, Bar and Shapes (B 133 – 54 T)

Spec. for Threadless Copper Pipe (B 302 – 56 T)

Spec. for Copper Drainage Tube (DWV) (B 306 – 56 T)

*Adopted as Standard, Revisions in:*

Spec. for Seamless Copper Pipe, Standard Sizes (B 42 – 55)

Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stocks (B 111 – 56)

Spec. for Seamless Copper Boiler Tubes (B 13 – 55)

Spec. for Seamless Red Brass Pipe, Standard Sizes (B 43 – 55)

Spec. for Seamless Copper Tube (B 75 – 55)

Spec. for Phosphor Bronze Wire (B 159 –54)

### *Committee B-6 on Die-Cast Metals and Alloys:*

Report presented by W. Babington, chairman, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Spec. for Aluminum-Base Alloy Die Castings (B 85 – 56 T)

Spec. for Zinc-Base Alloy Die Castings (B 86 – 53 T)

Spec. for Zinc-Base Alloys in Ingot Form for Die Castings (B 240 – 52 T)

*Adopted as Standard, Revisions in:*

Spec. for Magnesium-Base Alloy Die Castings (B 94 – 52)

Spec. for Copper-Base (Brass) Alloy Die Castings (B 176 – 52)

***Committee B-8 on Electrodeposited Metallic Coatings:***

Report presented in the absence of the chairman by J. W. Caum, and the following action taken:

*Accepted as Tentative:*

Rec. Practice for the Preparation of Lead and Lead Alloys for Electroplating (B 319 - 57 T)

***Committee B-9 on Metal Powders and Metal Powder Products:***

Report presented in the absence of the chairman by J. W. Caum, and accepted as a report of progress.

***Committee E-3 on Chemical Analysis of Metals:***

Report presented by Arba Thomas, chairman, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Methods for Chemical Analysis of White Metal Bearing Alloys (E 57 - 56 T)

*Adopted as Standard:*

Methods for Chemical Analysis of Antimony Metal (E 86 - 50 T)

*Adopted as Standard, Revisions in:*

Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 59 - 45)
Photometric Methods for Chemical Analysis of Copper and Copper-Base Alloys (E 62 - 56)

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## TWENTY-SECOND SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 19, 4:00 P.M.

SESSION CHAIRMAN: E. J. ALBERT

***Committee D-1 on Paint, Varnish, Lacquer and Related Products:***

Report presented by W. T. Pearce, chairman, and the following actions taken:

*Accepted as Tentative:*

Methods of Testing Fatty Acids Used in Protective Coatings (D 1467 - 57 T)
Test for Volatile Matter in Tricresylphosphate (D 1468 - 57 T)
Test for Total Rosin Acids Content of Coating Vehicles (D 1469 - 57 T)
Test for Two-Parameter 60-deg Specular Gloss (D 1471 - 57 T)
Test for Color-Difference Using the General Electric Spectrophotometer (D 1472 - 57 T)
Test for Oil Absorption of Pigments by Gardner-Coleman Procedure (D 1473 - 57 T)
Test for Knoop Indentation Hardness of Paint, Varnish, and Lacquer Coatings (D 1474 - 57 T)
Test for Density of Paint Type Materials (D 1475 - 57 T)
Test for Heptane Miscibility of Lacquer Solvents (D 1476 - 57 T)

*Accepted as Tentative, Revisions in:*

Test for Alcohol in Methyl Isobutyl Ketone (D 1362 - 55 T)
Test for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood (D 1211 - 52 T)
Test for Tinting Strength of White Pigments (D 332 - 55 T)
Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 52)
Methods of Testing Drying Oils (D 555 - 54)

*Adopted as Standard:*

Test for Effect of Household Chemicals on Clear and Pigmented Organic Finishes (D 1308 - 57 T)
Test for Acid Number of Bleach Lac and Bleached Lac Varnish (D 29 - 54 T) (Sections 6 to 13)

*Adopted as Standard, Revisions in:*

Def. of Terms Relating to Paint, Varnish, Lacquer and Related Products (D 16 - 52)
Spec. for Orange Shellac and Other Lacs (D 237 - 55)

Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 53)

*Tentatives Withdrawn:*

Test for Ester Value of Tricresyl Phosphate (D 268 - 49 T)

Test for Purity of Acetone and Methyl Ethyl Ketone (D 1154 - 54 T)

Through an inadvertence there had been included in the report of Committee D-1 the recommendation for the adoption as standard of Method D 714 - 54 T and Rec. Practice D 1006 - 51 T, and these recommendations accordingly were deleted.

### Committee D-11 on Rubber and Rubber-like Materials:

Report presented in the absence of the chairman by F. Y. Speight, and the following actions taken:

*Accepted as Tentative:*

Methods for Sampling and Sample Preparation of Synthetic Elastomers (Solid Styrene-Butadiene Copolymers) (D 1485 - 57 T), with extensive editorial changes.

Test for Indentation of Hard Rubber by Type D Durometer (D 1484 - 57 T)

*Accepted as Tentative, Revisions in:*

Method of Sample Preparation for Physical Testing of Rubber Products (D 15 - 55 T)

Spec. for Friction Tape for General Use for Electrical Purposes (D 69 - 52 T)

Spec. for Rubber Insulating Tape (D 119 - 48 T)

Methods of Testing Rubber Hose (D 380 - 51 T)

Method of Testing Hard Rubber Products (D 530 - 55 T)

Method of Testing Automotive Air Brake and Vaccum Brake Hose (D 622 - 55 T)

Methods of Testing Asphalt Composition Battery Containers (D 639 - 52 T)

Spec. for Heavy-Duty Neoprene Sheath for Wire and Cable (D 752 - 54 T)

Spec. for Light-Duty Neoprene Sheath for Wire and Cable (D 753 - 54 T)

Test for Viscosity of Rubber and Rubber-Like Materials by the Shearing Disk Viscometer (D 927 - 55 T)

Spec. for Ozone Resisting Rubber Insulating Tape (D 1373 - 55 T)

Method for Testing Synthetic Rubber Latices (Butadiene-Styrene Copolymers) (D 1417 - 56 T)

Test for Resistance to Light Checking and Cracking of Rubber Compounds (D 518 - 44)

Through an inadvertence the change in Tentative Method of Test D 412 - 51 T was set forth in the report as preprinted under the heading of Revision of Tentative whereas it should have been indicated as an editorial change.

*Adopted as Standard:*

Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (D 865 - 54 T)

### Committee D-14 on Adhesives:

Report presented in the absence of the chairman by F. W. Reinhart, and the following actions taken:

*Accepted as Tentative:*

Test for Amylaceous Matter in Adhesives (D 1488 - 57 T)

Test for Nonvolatile Content of Aqueous Adhesives (D 1489 - 57 T)

Test for Nonvolatile Content of Urea-Formaldehyde Resin Solutions (D 1490 - 57 T)

*Adopted as Standard:*

Test for Effect of Mold Contamination on Permanence of Adhesive Preparations and Adhesive Bonds (D 1286 - 53 T)

Test for Cross-Lap Specimens for Tensile Properties of Adhesives (D 1344 - 54 T)

Rec. Practice for Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives (D 904 - 46 T)

Rec. Practice for Determining Strength Development of Adhesive Bonds (D 1144 - 51 T)

### Committee D-18 on Soils for Engineering Purposes:

Report presented by E. J. Kilcawley, chairman, and the following actions taken:

*Adopted as Standard:*

Test for Bearing Capacity of Soil for Static Load on Spread Footings (D 1194 - 52 T)

Method of Repetitive Static Load Tests of Soils, for Use in Evaluation and Design of Airport and Highway Pavements (D 1195 - 52 T)

Method of Nonrepetitive Static Load Tests of Soils, for Use in Evaluation and Design of Airport and Highway Pavements (D 1196 - 52 T)

*Adopted as Standard, Revisions in:*

Test for Cement Content of Soil-Cement Mixtures (D 806 - 47) (Jointly with Committee D-4)
Test for Moisture-Density Relations of Soil-Cement Mixtures (D 558 - 44) (Jointly with Committee D-4)
Method of Wetting-and-Drying Test of Compacted Soil-Cement Mixtures (D 559 - 44) (Jointly with Committee D-4)
Method of Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures (D 560 - 44) (Jointly with Committee D-4)

Committee D-18 withdrew from the report as preprinted the new proposed Tentative Method for Thin-Walled Tube Sampling of Soils, and the new proposed Tentative Method for Penetration Test and Split-Barrel Sampling of Soils.

***Committee D-20 on Plastics:***

Report presented by F. W. Reinhart, chairman, and the following actions taken:

*Accepted as Tentative:*

Rec. Practice for Resistance of Plastics to Artificial Weathering Using Fluorescent Light and Fog Chamber (D 1501 - 57 T)
Test for Transverse Strength of Corrugated Reinforced Plastic Panels (D 1502 - 57 T)
Spec. for Extruded Cellulose Acetate Butyrate Pipe (D 1503 - 57 T)
Test for Orientation Release Stress of Plastic Sheeting (D 1504 - 57 T)
Test for Measurement of Density of Plastics by the Density-Gradient Technique (D 1505 - 57 T)

The committee withdrew from the report as preprinted the Proposed Tentative Spec. for Glass Fabric Reinforced Epoxy Resin Laminates.

*Accepted as Tentative, Revisions in:*

Test for Brittleness Temperature of Plastics and Elastomers by Impact (D 746 - 55 T)
Test for Water Absorption of Plastics (D 570 - 54 T)
Def. of Terms Relating to Plastics (D 883 - 56 T), with the withdrawal of the proposed new definition for Fabricating.
Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 - 56 T), with the withdrawal of the proposed revised definition of Pit.
Spec. for Phenolic Molding Compounds (D 700 - 55 T)
Test for Measuring Flow Rate of Thermoplastics by Extrusion Plastometer (D 1238 - 52 T)
Test for the Colorfastness of Plastics to Light (D 620 - 49)
Rec. Practice for Accelerated Weathering of Plastics Using S-1 Bulb and Fog Chamber (D 795 - 49)

The committee withdrew from the report as preprinted the recommendations for the revision of Tentative Spec. D 1201 - 52 T, and revision and reversion to tentative of Standard Spec. D 705 - 55.

*Adopted as Standard:*

Method of Testing Cellulose Acetate Butyrate (D 817 - 55 T) (Jointly with Committee D-23)
Test for Bursting Strength of Round Rigid Plastic Tubing (D 1180 - 51 T)
Test for Measuring the Molding Index of Thermosetting Molding Powder (D 731 - 55 T)
Spec. for Molds for Test Specimens of Plastic Molding Materials (D 647 - 54 T)
Spec. for Vinylidene Chloride Molding Compounds (D 729 - 52 T)

Through an inadvertence there had been included in the report as preprinted the recommendation for the adoption as standard of Tentative Method D 1181 - 51 T, and the recommendation accordingly has been deleted.

*Adopted as Standard, Revisions in:*

Test for Ammonia in Phenol-Formaldehyde Molded Materials (D 834 - 49)

The committee withdrew from the report as preprinted the recommendation for the revision of Standard Method D 648 - 56.

***Committee D-25 on Casein and Similar Protein Materials:***

Report presented by H. W. Shader, chairman, and accepted as a report of progress.

## TWENTY-THIRD SESSION—MARBURG LECTURE[10]

WEDNESDAY, JUNE 19, 4:30 P.M.

SESSION CHAIRMAN: PRESIDENT R. A. SCHATZEL

***Edgar Marburg Lecture:***

President Schatzel opened the session by giving a brief statement of the purpose of the lecture. Everett P. Partridge, Director, Hall Laboratories, Pittsburgh, Pa., was then introduced by the Executive Secretary R. J. Painter as the thirty-first Edgar Marburg Lecturer. Speaking on the subject, "Your Most Important Raw Materials—Water," Mr. Partridge discussed the fact that water is as basic to industry as it is to life itself. The problem is to catch it, store it, and transport it to the regions where we need it. Then we must use and re-use it efficiently. Like most raw materials, water is contaminated with various substances. Some of these impurities are the natural result of flow over or through the surface of the earth. More and more, however, the wastes from our human activities have complicated the re-use of water. What must be done to prepare water for industry depends on the ways it is to be used. Ultra-pure water containing no more than 50 parts per billion of total impurities may be specified for a once-through boiler, while the effluent from a municipal sewage plant serves satisfactorily for cooling equipment in a great steel mill.

President Schatzel expressed appreciation to Mr. Partridge for his excellent lecture and presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

## TWENTY-FOURTH SESSION—SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS[11]

THURSDAY, JUNE 20, 9:30 A.M.

SESSION CO-CHAIRMEN: C. C. WOOLSEY
G. D. CALKINS

### *I. Consideration of Radiation Effects in Design*

System's Consideration Effecting Selection of Structural Materials for Nuclear Power Plant Components—Walter L. Fleischmann, presented by the author.

Problems in Standardization of Techniques in Radiation Studies—M. J. Feldman and R. H. Fillnow, presented by Mr. Feldman

### *II. Radiation Facilities and Techniques*

Application of the Battelle Research Reactor to Radiation-Effects Studies—J. W. Chastain, Jr., presented by the author.

Examination and Testing Techniques at the HAPO Radio-Metallurgy Laboratory—C. L. Boyd, presented by the author.

(*Symposium Continued in the Twenty-Sixth Session*)

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[10] Issued as a separate publication.

[11] Issued as separate publication, *ASTM STP No. 220.*

## TWENTY-FIFTH SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 20, 11:30 A.M.

SESSION CHAIRMAN: J. H. KOENIG

### *Committee C-8 on Refractories:*

Report presented in the absence of the chairman by R. J. Painter, and the following actions taken:

*Accepted as Tentative:*

Classification of Castable Refractories (C 401 - 57 T)

*Accepted as Tentative, Revisions in:*

Method for Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 - 49)
Spec. for Fireclay-Base Castable Refractories for Boiler Furnaces and Incinerators (C 213 - 55)
Test for Disintegration of Refractories in an Atmosphere of Carbon Monoxide (C 288 - 56)
Classification of Fireclay Refractory Brick (C 27 - 56)

*Adopted as Standard, Revisions in:*

Classification of Insulating Fire Brick (C 155 - 47)

### *Committee C-11 on Gypsum:*

Report presented in the absence of the chairman by B. W. Nies, vice-chairman, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Spec. for Inorganic Aggregates for Use in Interior Plaster (C 35 - 54 T) (Jointly with Committee C-7)

*Adopted as Standard, Revisions in:*

Spec. for Gypsum Plasters (C 28 - 55)

### *Committee C-14 on Glass and Glass Products:*

Report presented in the absence of the chairman by R. J. Painter, and the following actions taken:

*Adopted as Standard:*

Test for Average Linear Expansion of Glass (C 337 - 54 T)
Test for Softening Point of Glass (C 338 - 54 T)

### *Committee C-16 on Thermal Insulating Materials:*

Report presented in the absence of the chairman by R. J. Painter, and the following actions taken:

*Accepted as Tentative:*

Test for Consistency of Wet-Mixed Thermal Insulating Cements (C 405 - 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Structural Insulating Board Made from Vegetable Fibers (C 208 - 55 T)
Methods of Testing Structural Insulating Board Made from Vegetable Fibers (C 209 - 55 T)

### *Committee C-21 on Ceramic Whiteware and Related Products:*

Report presented in the absence of the chairman by J. H. Koenig, and the following actions taken:

*Accepted as Tentative:*

Test for Compressive (Crushing) Strength of Fired Whiteware Materials (C 407 - 57 T)
Test for Thermal Conductivity of Ceramic Whitewares (C 408 - 57 T)

### *Committee C-22 on Porcelain Enamel:*

Report presented in the absence of the chairman by R. J. Painter, and the following actions taken:

*Accepted as Tentative:*

Test for Torsion Resistance of Laboratory Specimens of Porcelain-Enameled Iron and Steel (C 409 - 57 T)

*Accepted as Tentative, Revisions in:*

Def. of Terms Relating to Porcelain Enamel (C 286 - 56 T)

*Adopted as Standard:*

Test for Reflectivity and Coefficient of Scatter of White Porcelain Enamels (C 347 - 54 T)

***Committee D-24 on Carbon Black:***

Report presented by S. R. Doner, chairman, and the following actions taken:

*Accepted as Tentative:*

Test for Pour Density of Pelleted Carbon Black (D 1513 - 57 T), with the following change:

*Section 3.*—Add to the end of this section, "Precaution: Avoid jarring or vibrating the container in any way until after the excess has been struck off the top."

Test for Attrition of Pelleted Carbon Black (D 1507 - 57 T)

Test for Sieve Residue from Carbon Black (D 1514 - 57 T), with the following change:

*Table I.*—Add No. 100 sieve with the values, 149 micron sieve openings; wire diameter, in., min. 0.096; max. 0.125.

Test for Pellet Size Distribution of Carbon Black (D 1511 - 57 T)

Test for Fines Content of Pelleted Carbon Black (D 1508 - 57 T), with the deletion of Section 3(*g*).

Test for Ash Content of Carbon Black (D 1506 - 57 T)

Test for Heating Loss of Carbon Black (D 1509 - 57 T)

Test for pH Value of Carbon Black (D 1512 - 57 T)

Test for Iodine Adsorption Number of Carbon Black (D 1510 - 57 T)

## TWENTY-SIXTH SESSION—SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS[11]

(*Symposium Continued from Twenty-Fourth Session*)

THURSDAY, JUNE 20, 2:00 P.M.

SESSION CO-CHAIRMEN: C. C. WOOLSEY
G. D. CALKINS

### *III. Radiation Effect Studies of Materials*

Effect of Heat Treatment and Burnup on the Radiation Stability of Uranium-10 w/o Molybdenum Fuel Alloys—G. D. Calkins, J. E. Gates, F. A. Rough, D. O. Leeser and A. Del Grosso, presented by Mr. Calkins.

Engineering Effects of Radiation on Nuclear Fuels—B. Lustman, presented by the author.

A Survey of the Radiation Stability of Hydrocarbon Fuels—J. B. Carroll, R. O. Bolt, and J. A. Bert, presented by Mr. W. T. Stewart.

Selection of Organic Materials as Reactor Coolant-Moderators—E. L. Colichman and H. R. J. Gercke, presented by Mr. Gercke.

Effects of Radiation on Electronic Components.—I. Semiconductor Devices—M. A. Xavier, S. Nelson, A. Yefsky, A. Walters, and G. J. Rotariu, presented by Mr. Rotariu.

## TWENTY-SEVENTH SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 20, 4:30 P.M.

SESSION CHAIRMAN: R. W. SENIFF

***Committee A-10 on Iron Chromium, Iron-Chromium Nickel and Related Alloys:***

Report presented by Jerome Strauss, chairman, and the following actions taken; the revisions of the specifications which are under joint jurisdictions with Committee A-1 are subject to confirming letter ballot of Committee A-10:

*Accepted as Tentative:*

Spec. for Corrosion-Resisting Chromium-Nickel-Manganese Steel (Types 201 and 202) Plate. Sheet, and Strip (A 412 - 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 56 T) (Jointly with Committee A-1)

*Adopted as Standard:*

Rec. Practice for Descaling and Cleaning Stainless Steel Surfaces (A 380 - 54 T)

*Adopted as Standard, Revisions in:*

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 55) (Jointly with Committee A-1)
Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 56) (Jointly with Committee A-1)
Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 55) (Jointly with Committee A-1)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 56) (Jointly with Committee A-1)

### *Committee D-5 on Coal and Coke:*

Report presented by O. W. Rees, chairman, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Methods of Laboratory Sampling and Analysis of Coal (D 271 - 48)

*Adopted as Standard, Revisions in:*

Test for Free Swelling Index of Coal (D 720 - 46)

### *Committee D-7 on Wood:*

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Methods for Establishing Structural Grades of Lumber (D 245 - 49 T)
Spec. for Modified Wood (D 1324 - 54 T)

### *Committee D-12 on Soaps and Other Detergents:*

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

*Accepted as Tentative:*

Test for Copper in Soaps and Soap Products (D 460 - 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Trisodium Phosphate (D 538 - 55 T)
Def. of Terms Relating to Soaps and Other Detergents (D 459 - 56)

*Adopted as Standard:*

Spec. for Soda Ash (D 458 - 55 T)
Spec. for Sodium Metasilicate (D 537 - 55 T)
Test for Buffering Action of Metal Cleaners (D 1279 - 53 T)
Method of Total Immersion Corrosion Test of Soak Tank Metal Cleaners (D 1280 - 53 T)
Test for Rinsing Properties of Metal Cleaners (D 1281 - 53 T)
Method of Aerated Immersion Corrosion Test for Metal Cleaners (D 1374 - 55 T)

*Adopted as Standard, Revisions in:*

Methods of Sampling and Chemical Analysis of Alkaline Detergents (D 501 - 55)

### *Committee D-17 on Naval Stores:*

Report presented by V. E. Grotlisch, chairman, and the following actions taken:

*Adopted as Standard, Revisions in:*

Methods of Sampling and Testing Turpentine (D 233 - 51)
Methods of Testing Dipentene (D 801 - 48)
Methods of Sampling and Testing Pine Oil (D 802 - 49)
Def. of Terms Relating to Naval Stores and Related Products (D 804 - 52)

### *Committee D-21 on Wax Polishes and Related Materials:*

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

*Adopted as Standard:*

Test for Total Ash and Silica in Water-Emulsion Waxes (D 1288 - 53 T)
Test for Sediment in Water-Emulsion Waxes by Centrifuge (D 1290 - 53 T)
Test for Paraffin-Type Hydrocarbons in Carnauba Wax (D 1342 - 56 T)

### *Committee E-2 on Emission Spectroscopy:*

Report presented by D. L. Fry, chairman, and the following actions taken:

*Accepted as Tentative:*

Method for Spectrochemical Analysis of Nickel Alloys by the Powder—D-C Arc Technique (E 129 - 57 T)
Rec. Practice for Designation of Shapes and

Sizes of Preformed Graphite Electrodes (E 130 - 57 T)

*Accepted for Publication as Information Only:*

Suggested Method for Spectrochemical Analysis of Nickel-Base High-Temperature Alloys by the Point-to-Plane Spark Technique

Suggested Method for Spectrochemical Analysis of Bismuth-Cadmium Eutectic Alloy by the Powder—D-C Arc Technique

Suggested Method for Spectrochemical Analysis of Tin-Cadmium Eutectic Alloy by the Powder—D-C Arc Technique

Suggested Method for Spectrochemical Analysis of Aluminum and Its Alloys by the Point-to-Plane Spark and Intermittent Arc Techniques, Using Recording Photoelectric Spectrometer

Suggested Method for Spectrochemical Analysis of Magnesium and Its Alloys by the Pin-to-Pin Spark Technique

Suggested Method for Spectrochemical Analysis of Basic Open-Hearth Slags for Lime-Silica Ratios by the Powder-Spark Technique

Suggested Method for Spectrochemical Analysis of Alumina Ceramic Materials by the Powder—D-C Arc Technique

Suggested Method for Spectrochemical Analysis of Cobaltic Oxide for Impurities by the Powder—D-C Arc Technique

### *Committee E-13 on Absorption Spectroscopy:*

Report presented in the absence of the chairman by R. F. Robey, secretary, and the following action taken:

*Accepted as Tentative:*

Def. of Terms and Symbols Relating to Absorption Spectroscopy (E 131- 57 T)

## TWENTY-EIGHTH SESSION—SYMPOSIUM ON DETERMINATION OF DISSOLVED OXYGEN IN WATER[12]

THURSDAY, JUNE 20, 8:00 P.M.

SESSION CHAIRMAN: F. U. NEAT

### *Committee D-19 on Industrial Water:*

Report presented by Max Hecht, chairman, and the following actions taken:

*Accepted as Tentative:*

Methods of Sampling Homogeneous Industrial Waste Water (D 1496 - 57 T)

Test for Iron in High-Purity Industrial Water (D 1497 - 57 T)

The committee withdrew from the report as preprinted the recommendation for the publication as tentative of the Method of Test for Appearance Properties of Industrial Water and Industrial Waste Water.

*Accepted as Tentative, Revisions in:*

Test for Acidity and Basicity (Alkalinity) in Industrial Water and Industrial Waste Water (D 1067 - 55 T)

Test for Hydrazine in Industrial Water (D 1385 - 55 T)

Test for Nitrite Ion in Industrial Water (D 1254 - 53 T)

*Adopted as Standard:*

Test for Total Carbon Dioxide and the Calculation of the Carbonate and Bicarbonate Ions in Industrial Water (D 513 - 54 T)

Test for Chlorine Requirements of Industrial Water and Industrial Waste Water (D 1291 - 53 T)

Test for Fluoride Ion in Industrial Water and Industrial Waste Water (D 1179 - 55 T)

Test for Hardness in Industrial Water (D 1126 - 55 T)

Test for Residual Chlorine in Industrial Water (D 1253 - 53 T)

Method for Determination of Thickness of Internal Deposits on Tubular Heat Exchange Surfaces (D 1341 - 54 T)

*Adopted as Standard, Revisions in:*

Def. of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 - 56)

*Accepted for Publication as Information Only:*

Non-Referee Method of Test for Acidity and Alkalinity in Industrial Water

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[12] Issued as separate publication *ASTM STP No. 219.*

***Papers:***

Introduction by F. U. Neat.

A Study of the Accuracy of Methods of Testing for Dissolved Oxygen in High Purity Water—K. G. Stoffer, presented by the author.

Polarographic Measurement of Dissolved Oxygen—W. W. Eckenfelder, Jr., and Brother Conrad Burris, presented by Mr. Burris.

The Beckman Oxygen Analyzer—Thomas Finnegan and Ross C. Tucker, presented by Mr. Finnegan.

Evaluation of Hartmann and Braun Dissolved Oxygen Recorder—A. J. Ristaino and A. A. Dominick, presented by Mr. Ristaino.

Determination of Dissolved Oxygen by Means of a Cambridge Analyzer—H. A. Grabowski, presented by the author.

## TWENTY-NINTH SESSION—MASONRY

FRIDAY, JUNE 21, 10:30 A.M.

SESSION CHAIRMAN: M. N. CLAIR

Measuring Shrinkage of Concrete Block (Comparison of Test Methods)—E. L. Saxer and H. T. Toennies, presented by Mr. Saxer.

Recent Disintegration of Mortar in Brick Walls—C. C. Connor and W. E. Okerson, presented by Mr. Connor.

Fallacies in the Current Per Cent of Total Absorption Method for Determining and Limiting the Moisture Content of Concrete Block—Carl A. Menzel, presented by the author.

## THIRTIETH SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 21, 12:30 P.M.

SESSION CHAIRMAN: A. A. BATES

***Committee A-3 on Cast Iron:***

Report presented by D. E. Krause, chairman, and the following actions taken:

*Adopted as Standard:*

Spec. for Cast Iron Pressure Pipe (A 377 - 54 T)

*Adopted as Standard, Revisions in:*

Spec. for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (A 278 - 56)

***Committee A-5 on Corrosion of Iron and Steel:***

Report presented by Marc Darrin, chairman, and the following actions taken:

*Accepted as Tentative:*

Spec. for Zinc-Coated (Galvanized) Low-Carbon Steel Armor Wire (A 411 - 57 T)

*Adopted as Standard, Revisions in:*

Spec. for Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 - 48)

Spec. for Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 - 48)

***Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts:***

Report presented by E. I. Shobert, II, chairman, and the following actions taken:

*Adopted as Standard, Revisions in:*

Method of Accelerated Life Test for Metallic Materials for Electrical Heating (B 76 - 39): covering (1) adoption as standard of the revised tentative revision; and (2) the immediate adoption of revisions of the Appendix.

Spec. for Drawn or Rolled Alloy, 80 Per Cent

Nickel, 20 Per Cent Chromium, for Electrical-Heating Elements (B 82 - 52)

***Committee C-1 on Cement:***

Report presented by R. R. Litehiser, chairman, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Spec. for Portland Blast-Furnace Slag Cement (C 205 - 56 T)
Spec. for Flow Table for Use in Tests of Hydraulic Cement (C 230 - 55 T)
Test for Bleeding of Cement Pastes and Mortars (C 243 - 55 T)
Spec. for Portland Pozzolan Cement (C 340 - 55 T)
Spec. for Slag Cement (C 358 - 55 T)
Methods of Chemical Analysis of Portland Cement (C 114 - 53)

*Adopted as Standard:*

Spec. for Masonry Cement (C 91 - 56 T)

***Committee C-12 on Mortars for Unit Masonry:***

Report presented by R. E. Copeland, chairman, and the following actions taken:

*Accepted as Tentative:*

Spec. for Aggregates for Masonry Grout (C 404 - 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Mortar for Unit Masonry (C 270 - 54)

***Committee C-15 on Manufactured Masonry Units:***

Report presented by J. W. Whittemore, chairman, and the following actions taken:

*Adopted as Standard, Revisions in:*

Spec. for Building Brick (C 62 - 50)
Spec. for Facing Brick (C 216 - 50): covering (1) adoption as standard of the tentative revision of Section 4 and Explanatory Notes; and (2) the immediate adoption of revisions of Section 3.
Spec. for Structural Clay Load-Bearing Wall Tile (C 34 - 55)
Spec. for Structural Clay Non-Load-Bearing Tile (C 56 - 52)
Spec. for Structural Clay Floor Tile (C 57 - 52)
Methods of Sampling and Testing Brick (C 67 - 50)

***Advisory Committee on Corrosion:***

Report presented in the absence of the chairman by S. F. Etris, secretary, and accepted as a report of progress.

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## THIRTY-FIRST SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 21, 12:30 P.M.

SESSION CHAIRMEN: R. C. ALDEN
PRESIDENT R. A. SCHATZEL

***Committee D-4 on Road and Paving Materials:***

Report presented in the absence of the chairman by A. B. Cornthwaite, vice-chairman, and the following actions taken:

*Accepted as Tentative, Revisions in:*

Test for Moisture-Density Relations of Soils (D 698 - 42 T) (Jointly with Committee D-18)
Spec. for Crushed Stone, Crushed Slag, and Gravel for Single or Multiple Bituminous Surface Treatment (D 1139 - 55 T)
Spec. for Crushed Stone, Crushed Slag, and Gravel for Water-Bound Macadam Base and Surface Courses of Pavements (D 694 - 55)
Spec. for Mineral Filler for Sheet Asphalt and Bituminous Concrete Pavements (D 242 - 39)
Test for Soft Particles in Coarse Aggregates (C 235 - 54) (Jointly with Committee C-9)

*Adopted as Standard:*

Test for Bitumen Content of Paving Mixtures by Centrifuge (D 1097 - 54 T)

*Adopted as Standard, Revisions in:*

Spec. for Emulsified Asphalt (D 977 – 53)
Test for Cement Content of Soil-Cement Mixtures (D 806 – 47) (Jointly with Committee D-18)
Test for Moisture-Density Relations of Soil-Cement Mixtures (D 558 – 44) (Jointly with Committee D-18)
Method of Wetting-and-Drying Test of Compacted Soil-Cement Mixtures (D 559 – 44) (Jointly with Committee D-18)
Method of Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures (D 560 – 44) (Jointly with Committee D-18)

The last three recommendations appear only in the report of Committee D-18 but they are included here since they have been approved by letter ballot action of Committee D-4.

### Committee D-6 on Paper and Paper Products:

Report presented by R. H. Carter, chairman, and the following actions taken:

*Adopted as Standard, Revisions in:*

Test for Reducible Sulfur in Paper (D 984 – 50), with the additional change of deleting in Section 6, line 21, the word "prepared."

### Committee D-9 on Electrical Insulating Materials:

Report presented in the absence of the chairman by J. E. Gibbons, secretary, and the following actions taken:

*Accepted as Tentative:*

Test for Set Time of Thermosetting Phenol Formaldehyde Varnishes (D 1472 – 57 T)
Test for 2,6 Ditertiary-Butyl Para-Cresol in New Electrical Insulating Oils (D 1473 – 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Natural Muscovite Mica Based on Visual Quality (D 351 – 53 T)
Methods of Testing Pressure-Sensitive Tapes Used for Electrical Insulation (D 1000 – 55 T)
Rec. Practice for Purchase of Uninhibited Mineral Oil for Use in Transformers and Circuit Breakers (D 1040 – 54 T)
Methods of Testing Sheet and Plate Materials Used for Electrical Insulation (D 229 – 49)
Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 48)

*Adopted as Standard:*

Methods of Testing Silicone Varnishes (D 1346 – 56 T)

*Accepted for Publication as Information Only:*

Method for Corona Measurement
Test for Dust and Fog Tracking Resistance of Electrical Insulating Materials

There had been included in the preprinted report of Committee D-9 the joint recommendation with Committee D-20 for the publication as tentative of Specifications for Glass Fabric Reinforced Epoxy Resin Laminates. This recommendation was withdrawn by Committee D-20 and accordingly publication of the specifications will be withheld.

### Committee D-15 on Engine Antifreezes:

Report presented by R. E. Vogel, chairman, and accepted as a report of progress.

### Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials:

Report presented by D. F. Gould, chairman, and the following actions taken subject to letter ballot approval of Committee D-16:

*Accepted as Tentative:*

Test for Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration (D 1491 – 57 T)
Test for Bromine Index of Aromatic Hydrocarbons by Coulometric Titration (D 1492 – 57 T)
Test for Solidification Points of Chemicals (D 1493 – 57 T)

*Accepted for Publication as Information Only:*

Method for the Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene

### Committee F-1 on Materials for Electron Tubes and Semiconductor Devices:

Report presented in the absence of the chairman by P. J. Smith, and the following actions taken:

*Accepted as Tentative:*

Spec. for Clear Nickel-Clad and Nickel-Plated Steel Strip for Electron Tubes (F 1 – 57 T)
Spec. for Aluminum-Clad Steel Strip and Nickel-Steel-Aluminum Composite Strip for Electron Tubes (F 2 – 57 T)

Spec. for Clear Nickel Strip for Electron Tubes (F 3 - 57 T)
Spec. for Carbonized Nickel Strip and Carbonized Nickel-Plated and Nickel-Clad Steel Strip for Electron Tubes (F 4 - 57 T)

*Accepted as Tentative, Revisions in:*

Spec. for Circular Cross-Section Nickel Cathode Sleeves for Electronic Devices (F 239 - 49 T)

At this point Mr. Alden turned over to Dr. Schatzel the chairmanship of the session.

### Committee D-2 on Petroleum Products and Lubricants:

Report presented by Harold M. Smith, chairman, and the following actions taken:

*Accepted as Tentative:*

Test for Low-Temperature Torque of Ball Bearing Greases (D 1478 - 57 T)
Test for Measuring the Color of Petroleum Products (ASTM Color Scale) (D 1500 - 57 T)
Test for Freezing Point of Aviation Fuels (D 1477 - 57 T)
Test for Emulsion Stability of Soluble Cutting Oils (D 1479 - 57 T)
Test for Density and Specific Gravity of Viscous Materials and Melted Solids by Bingham Pycnometer Method (D 1480 - 57 T)
Test for Density and Specific Gravity of Hydrocarbon Liquids by Lipkin Bicapillary Viscous Oil Pycnometer (D 1481 - 57 T)
Test for Blocking Point of Paraffin Wax (D 1465 - 57 T)

*Accepted as Tentative, Revisions in:*

Test for Water and Sediment by Means of Centrifuge (D 96 - 52 T)
Test for Existent Gum in Fuels by Jet Evaporation (D 381 - 56 T)
Test for Distillation of Plant Spray Oils (D 447 - 55 T)
Test for Chlorine in Lubricating Oils and Greases by the Bomb Method (D 808 - 52 T)
Spec. for Aviation Gasolines (D 910 - 56 T)
Method of Gaging Petroleum and Petroleum Products (D 1085 - 56 T), with editorial changes.
Rec. Practice for Volume Calculations and Corrections in the Measurement of Petroleum and Petroleum Products (D 1087 - 52 T)
Test for Bromine Number of Petroleum Distillates (Color-Indicator Method) (D 1158 - 55 T)
Test for Bromine Number of Petroleum Distillates (Electrometric Method) (D 1159 - 55 T)
Test for Reduced Pressure Distillation of Petroleum Products (D 1160 - 56 T)
Methods of Calibrating Liquid Containers (Upright Tanks) (D 1220 - 56 T)
Test for Sulfur in Petroleum Products and Liquefied Petroleum Gases by the $CO_2$—$O_2$ Lamp Method (D 1266 - 55 T), with editorial changes.
Test for Chlorine in Lubricating Oil (Sodium Alcoholate Volumetric Method) (D 1317 - 54 T)
Test for Tensile Strength of Paraffin Wax (D 1320 - 54 T)
Test for Needle Penetration of Petroleum Waxes (D 1321 - 55 T)
Test for Estimation of Heat of Combustion of Liquid Petroleum Products (D 1405 - 56 T)
Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (D 1019 - 56 T)
Test for Heat of Combustion of Liquids by Bomb Calorimeter (D 240 - 50), with the following additional changes:

*Section 5.*—Delete this section, and renumber the subsequent sections accordingly.

*Section 9.*—Renumber as Section 8, and add the following Note 8 to the end of paragraph (*a*):

NOTE 8.—All mercury-in-glass thermometers must be corrected for scale error, using data from the thermometer certificate prescribed in Appendix I, Section A5(*a*) or (*b*). Beckmann thermometers also require a setting correction and an emergent stem correction (Appendix II, Section A12(*b*)). Solid-stem ASTM thermometers 56 F and 56 C do not require emergent stem corrections if all tests, including standardization, are performed within the same 10 F (5.5 C) interval. If operating temperatures exceed this limit, a differential emergent stem correction (Appendix II, Section A12(*a*)) must be applied to the corrected temperature rise, *t*, in all tests, including standardization.

NOTES 9 AND 10.—Delete these notes.

Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method) (D 873 - 49)
Test for Sulfated Residue from New Lubricating Oils (D 874 - 55)

*Adopted as Standard, Revisions in:*

Test for Melting Point of Paraffin Wax (D 87 - 42)
Test for Flash and Fire Points by Cleveland Open Cup (D 92 - 56)
Test for Cloud and Pour Points (D 97 - 47), with the following additional change:

NOTE 2.—Add to the end of the note, "Drying and filtering should be done always at a temperature at least 25 F above the approximate cloud point but otherwise not in excess of 120 F. Remove moisture, if present, by any suitable method, as by filtration through dry lintless filter paper until the oil is perfectly clear, but make such filtration at a temperature at least 25 F above the approximate cold point."

Methods of Analysis of Lubricating Greases (D 128 - 47)
Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method (D 129 - 52)
Def. of Terms Relating to Petroleum (D 288 - 53)
Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity (D 666 - 53)
Test for Water Tolerance of Aircraft Fuels (D 1094 - 53)

*Tentatives Withdrawn:*

Test for Sulfur in Petroleum Products by the Lamp-Gravimetric Method (D 90 - 55 T)
Test for Color of U. S. Military Gasoline, Automotive (Combat), by Means of an ASTM Color Standard (D 976 - 48 T)

*Accepted for Publication as Information Only:*

Method for Analysis of Certain Greases for Which Method D 128 - 47 Is Unsuitable
Method of Test for Particle Size Range of Graphite
Method for 20 Deg Specular Gloss of Waxed Paper
Spec. for Aviation Turbine Fuels
Revisions of Standard Method of Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 - 56)
Revisions of Standard Method of Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 - 55)
Revisions to Appendices of 1956 Manual for Rating Motor Fuels by Motor and Research Methods
Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene
Method of Test for Vanadium in Residual Fuel Oil, with editorial changes.
Method of Test for Carbon Number Distribution of Aromatic Compounds in Naphthas by Mass Spectrometry
Method of Test for Refractive Index of Viscous Liquids and Melted Solids, with editorial changes.
Method of Test for Distillation of Petroleum Products
Spec. for Distillation Equipment
Summary of Data Collected in the Cooperative Testing of Method D 240, Test for Heat of Combustion of Liquids by Bomb Calorimeter
Method of Test for Odor of Petroleum Wax
Supporting Data for Extension of Standard Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity (D 666 - 53) to Permit Conversion of Kinematic to Saybolt Furol Viscosities at 210 F

The committee withdrew from the report as preprinted the recommendations for publication as tentative of Method of Test for Thermal Stability of Fuel Oils, and publication as information only of the Test for Sulfur in Petroleum Products by the Rapid, High-Temperature Combustion Method. The recommendation with respect to Tentative Method of Test D 1019 - 56 T was changed from "Adoption of Tentative as Standard with Revisions" to "Revision of Tentative."

### *Closing Remarks:*

President Schatzel referred briefly to some of the activities of the last year and touches upon some of the plans for the future. After expressing appreciation for the cooperation he felt he had received on every hand and bespeaking the same for his successor he called upon Past-President K. G. Mackenzie to escort President elect R. C. Kropf to the platform. Upon receiving the emblems of the office, he pledges his efforts in carrying forward the work during the coming year.

EDITORS' NOTE.—The recommendations affecting standards referred to letter ballot were approved when the ballot was canvassed on September 16.

# SUMMARY OF PROCEEDINGS OF PHILADELPHIA SPRING MEETING

During the 1957 Committee Week held in Philadelphia, Pa., a special technical session was held on Wednesday, February 6, constituting the 1957 Spring Meeting:

## SYMPOSIUM ON THERMAL CONDUCTIVITY MEASUREMENTS AND APPLICATIONS OF THERMAL INSULATIONS

This symposium was sponsored by Committee C-16 on Thermal Insulating Materials. The Special Symposium Subcommittee was headed by Henry E. Robinson, National Bureau of Standards. C. B. Bradley, Johns-Manville Corp., acted as Chairman of the Symposium session.

The guarded hot plate apparatus for determining the thermal conductivity of insulating, building, and other materials, was the subject of review in four of the seven papers presented at the Symposium. A three-year research project on the effect of moisture on thermal insulation prompted the presentation of a fifth paper. Two additional papers, on the testing of underground piping thermal insulation, and stress corrosion cracking of insulated austenitic stainless steels, completed the symposium. All papers, with the exception of the paper on stress corrosion cracking by A. W. Dana, Jr., E. I. du Pont de Nemours & Co., Inc., which was published in the ASTM BULLETIN, No. 225, were issued as *ASTM Special Technical Publication No. 217* entitled "Symposium on Thermal Conductivity Measurements and Applications of Thermal Insulations."

The following papers with discussion are included in *STP 217*:

Introduction—Henry E. Robinson
An Improved Guarded Hot Plate Thermal Conductivity Apparatus with Automatic Controls—Zeno Zabawsky
The Use of Envelope Type Cold Plates in Thermal Conductivity Apparatus—C. F. Gilbo
Errors in Thermal Conductivity Measurement by the Guarded Hot Plate Due to Guard Ring Unbalances—W. Woodside and A. G. Wilson
Analysis of Errors Due to Edge Heat Loss in Guarded Hot Plates—William Woodside
Thermal Conductivty of Insulation Containing Moisture—F. A. Joy
Criteria for Testing Underground Piping Thermal Insulation—D. D'Eustachio

# OUR EXPANDING TECHNOLOGY AND ASTM

## ANNUAL ADDRESS BY THE PRESIDENT

RUDOLPH A. SCHATZEL[1]

JUNE 18, 1957

During the past six months I have had the great privilege and stimulating experience of visiting all our District Councils except one—the Ohio Valley District. We however, visited three other sections of the country where there are no districts, but where interest in ASTM is very much alive.

Each year at this time a short period is provided for your President to sing his "swan song" and then resume his place in the ranks to work for the Society under a new leader. That I pledge most willingly to do. I do not have any stirring message to bring you to repay you for the honor you have done me these many years and particularly this last, as I have served as your President. I should like to talk to you a few moments about our visit and present to you some thoughts on our Society.

As we traveled from District to District I was greatly impressed by the tremendously varied technologies, with the virility of interest and appreciation of our task in ASTM, the desire to advance the interest of our Society, and the loyalty and pride of so many who have served the Society so well, so long. Our forty and fifty-year members, Past-Presidents, honorary members, and committee members now long retired were on hand to advise and eager to hear of the Society's plans and accomplishment. At times they gently chided us about some pet project which is not moving with appropriate speed. These men and our committees are our Society, and we can all be justly proud.

Our visit to the unique Hot Metallurgical Laboratory of the Hanford Atomic Energy plant in Washington, and attendance later as your honored representative at the Nuclear Congress left an unforgettable memory and realization of the great strides and rapid changes in our technology.

The immediacy of our task became apparent from our discussions at Hanford. The questions were, how can we fit into ASTM better? and can ASTM do more to help us on our problems? Fortunately we have many committees studying the latter question and an over-all committee coordinating activities in the nuclear field. Whether we were inspecting models of the various dams designed to harness the rivers of the world, or attending meetings of over 400 men in one of our committees discussing mass spectrometry, the contribution of our Society in the development of new technologies was most evident and inspiring.

### THE NEED FOR BETTER COMMUNICATION

During our travels we also came in contact with many who are not members of ASTM who sometimes shocked us with their lack of knowledge of what ASTM is, and does. The following are typical of some of the questions asked:

1. How does ASTM affect our community? This I attempted to answer in my talk in Los Angeles,[2] since the roads

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[1] Vice-president and director of engineering, Rome Cable Corp., Rome, N. Y.

[2] R. A. Schatzel, "Where Do We Go from Here?" ASTM BULLETIN, No. 217, Oct. 1956, p. 21.

they travel on, the water they drink, their houses, and the clothes they wear—are all affected by ASTM research and standards.

2. "What is ASTM?" This question was asked by a state secretary of a leading professional engineering group, at a large dinner on Engineers' Day—a very charming and intelligent gentleman.

3. "Does ASTM develop Standards? I thought that was done by the American Standards Assn." This was asked by an English scientist visiting this country and very much concerned with standardization in England. When shown the statement in the constitution of ASA that its purpose is to stimulate the work of existing organizations and to encourage the establishment of organizations if none exist, but not to formulate standards, he said, "Well, well!"

4. A visiting French scientist with considerable acquaintance in America in the electrical industry said he thought standards in America were developed by the National Bureau of Standards. When shown a recent statement by the Bureau that it had representation on 408 committees of ASTM, he became very interested in our methods and organization.

There were many other similar questions, some quite amusing. I believe, however, that it is fair to say that we do need some better communication with many groups affected by our work, but not directly concerned. Steps to develop, greater understanding of our operation are being taken.

### ASTM AND THE GOVERNMENT

As indicated by the questions of those outside our borders, the relationship of ASTM to Government standardization, and to other professional and standardizing bodies in the United States is hard for them to understand. Our system of standardization is inherently tied to our system of free enterprise. It is a completely voluntary system, in which our Government may have representatives on committees, often acting as chairmen, and as at present, in our Society, members of government agencies serve on our Board of Directors. As a most important customer, the Government's representatives have great weight, but the Government does not try to dominate our work nor that of any other standardizing group on which it has representation.

It is also a fact that no single agency acts for the Government.

Although as far as we know, there is no single agency established on a national level to coordinate the needs of the Federal Government with those of industry as far as standards are concerned, ASTM has for many years, in fact, from its very inception, cooperated closely with those Governmental agencies that develop standards.

For many years we have had a Government Contacts Committee, and many of our committees have contact liaison with governmental departments, cooperating with Federal agencies such as General Services Administration and the Department of Defense, to avoid duplication and to obtain a better coordination between Government and industry standards.

An outstanding example of this kind of accomplishment is Federal Standard 791, which now merely refers by ASTM designation to about 90 test methods for petroleum products and lubricants. These were developed by our Committee D-2. In addition, our Committees on Plastics, Paints, Steel, Textiles, and Cement are actively helping to coordinate Federal and ASTM Standards. We also have a contract with the GSA to supply our ASTM Standards to the Government.

When we realize the great savings these programs effect in cost and manpower during peacetime, and the fact that time will not be available during an emergency for their development, this cooperative effort represents both a patriotic and an economic accomplishment for intelligent constructive cooperation—particularly on the part of our Government.

There are many similar examples, in other fields, of cooperation.

## ASTM AND OTHER SOCIETIES

In our relations with other professional and technical associations we are equally fortunate. In the field of standardization the American Society for Testing Materials is the oldest, in existence before the Bureau of Standards was organized, and one of the sponsors and organizers of the American Standards Assn. in 1918. It is not surprising, therefore, that approximately 250 technical societies and trade associations have representation on our technical committees. We also have formed a great many joint committees with other associations to conduct research with highly specialized groups in a particular field of scientific endeavor. This combination of skills and organizations results in a more rapid absorption of scientific research into an organized body of knowledge and the creation of new technologies as a contribution to our economy. The strength of ASTM derives from the fact that it serves the intelligent self interest of industry, Government and the consuming public.

Occasionally, a trade association or other group may find an apparent duplication of their efforts in our committees' work but more often they will bring the results of their work to our committees to subject them to the broader scrutiny of the consumer and general interests which are represented on all our committees. When duplication is found, it is either resolved by our appropriate Administrative Committee or by the formation of a Joint Committee.

There is one important guiding principle in all our work. Our committees are autonomous within their scope. Only when there is an expressed request to the committee and approval by the Society of its need, is a new field of endeavor considered. Only then when it does not duplicate the work of some other well qualified group, is the work authorized. Manpower and the tremendous needs of our technological development dictate cooperation rather than competition.

Since many organizations develop standards of various kinds and promulgate codes, there is a need for a voluntary central clearing house, so that in international standardization there is a unified American voice. This is the function of the American Standards Assn. Approximately one-third of the standards of ASA are the responsibility of ASTM. This is an example of teamwork and the avoidance of duplication to conserve technical manpower.

## THE JOB AHEAD

What of the job ahead; and how are we prepared to cope with it? Standardization is the result of research, and as one of our Directors has so aptly said, "Research is what makes our standards dynamic," moving and growing as new knowledge is developed. Standards, then, are the vehicle by which new scientific knowledge and its applications are assimilated in an orderly manner into our economy.

In 1955 Dr. Vannevar Bush, who headed the Office of Scientific Research and Development during wartime, testified as follows:

> But it would be a mistake to assume that the greatest result of the war from the standpoint of . . . application of science . . . lies in the appearance of . . . automation . . . we have new fibers, new plastics, and as a result a whole new industry. We have thermionic tubes, transistors, phosphors, and their application in radio, radar, television, and many other devices. We have greatly extended synthetic chemistry . . . we have

atomic power in the offing, and we may also have solar power . . . what is new is an accelerated pace in the application of scientific results in an economic manner.

We could also add that another new factor is the ready acceptance by the public of these techniques and materials. There is a noticeable quickening of pace—a lessening in the induction period from laboratory experiment to commercial channels that has resulted in the development of whole new industries which have a vested interest in change, of which automation is an example. In addition, we have new environments of vital importance. Among these are radiation, aerodynamic heating, high-intensity sound, broad-band shock and vibration, and combinations of these. We must have reliable data and methods of evaluation of the performance of materials under these new conditions. The cooperative resources of both industry and Government are necessary to provide the answers. Our Society has a most important part to play, and time is of the essence.

In addition to unusual applications and services, it is astounding to think of the volume of uses anticipated for some of our more prosaic materials. These also require sound standards. Our Vice-President, Kenneth Woods, who is chairman of the Highway Research Board, points out that the Bureau of Public Roads estimates that 49 million tons of steel, 1 billion 339 million barrels of cement, 128 million tons of bituminous material, and large quantities of other construction materials will be needed in the Federal highway construction program. On the New Jersey Turnpike alone, over 100 ASTM Standards were involved in the materials used. Whether for unique application or for large volume use, ASTM standards are an effective factor in purchase and control.

Many of our committees are already concerned with phases of these and other problems which mark the explosion of our technological progress. A detailed listing of our new committees, formed each year, and the number and diversity of the symposia held at each of our Annual and Spring meetings, catalog much of this progress.

## ASTM PLANS FOR THE FUTURE

Is ASTM prepared to play its part in this expanding development of scientific knowledge and technological advance? The rapid growth of ASTM measured by membership, standards, publications, and sound finance is a clear indication that the Society is more than keeping pace with the technological revolutions that are taking place in this country and the world at large. The sound and orderly growth of our Society is due to its contributions to our economy and technology in the past. A major problem of the future is to assure that these proliferations do not weaken our capacity for service and sound guidance, and that they are organized so that the work of the Society will continue at a high efficiency. To this end, a planning committee to examine the organization, procedures and operation of the Society has been authorized and is being formed. We cannot plan for the next sixty years or for the year 1999, but we can be assured that ASTM is prepared to continue the service to our industry and our country, which has typified its past.

In closing, may I again express my sincere appreciation to all the members of the Board who have served with me and to all of our wonderful Staff for their loyalty to the Society—and to you, for the great honor you have done me. I know that the same support is assured our new President in the year of progress which is immediately before us. As I take my place in the rank I do so with a profound sense of gratitude and of confidence in our Society's future.

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WHETHER American industry or any of its divisions are concentrating on war-time or peace-time problems, a better knowledge of materials—and still better materials—are important if progress is to be made at all rapidly. One very notable trend in recent years is the rapidly increasing tempo of our technology.

*HIGH LIGHTS, 1956 FINANCIAL OPERATIONS*

| | 1956 | 1955 | 1954 |
|---|---|---|---|
| *Gross Receipts:* | | | |
| Membership Dues | $269 000 | $252 100 | $236 400 |
| Sales of Publications | 695 950 | 521 500 | 470 500 |
| Other | 117 600 | 78 200 | 87 700 |
| Total | $1 082 550 | $851 800 | $794 600 |
| *Expenditures:* | | | |
| Salaries | $309 400 | $305 400 | $276 900 |
| Printing | 342 300 | 350 000 | 337 500 |
| Other | 244 800 | 196 000 | 196 600 |
| Total | $896 500 | $851 400 | $811 000 |
| Favorable Operating Balance | $186 050 | $400 | ... |
| Operating Deficit | ... | ... | $16 400 |
| *Net Assets:* | | | |
| Real Estate | $269 450 | $277 700 | $295 200 |
| Investments and Cash | $1 048 315 | 880 500 | 797 000 |
| Equipment | 32 900 | 29 500 | 29 700 |
| Total | $1 350 665 | $1 187 700 | $1 121 900 |
| Asset Equity of Each Member | $160 | $147 | $145 |
| Number of Members | 8 408 | 8 079 | 7 762 |
| | 1956 | 1955 | 1954 |
| Pages of Publications | 10 540 | 18 742 | 8 088 |
| Number of Employees | 75 | 69 | 65 |

Be the problem one of automation, or increased efficiency in the electrical generating field, or the construction of a plant to use nuclear energy, satisfactory materials are a critical part of the operation.

To ensure profitable operations, industry has become increasingly cost conscious. This means using the right materials efficiently. Careful analysis in many areas indicates that much more is being and still more can be done to use some of the newer families of products to cut costs materially.

All of this means increasing activity in our Society, concentrating as it does in the field of materials. Our research and standardization work grows in importance and volume. We are hard pressed to provide the authoritative information needed in so many fields and to provide the standard methods of test and specifications for evaluating and procuring quality materials which will do the tasks required.

Much progress has been made during the past year. We can cite here only a few of the newer projects. Only through continuing review of the ASTM BULLETIN, with its articles on committee meetings and new activities in existing committees, and by a careful review of the annual reports of the technical committees which carry out our work, can one hope to get a reasonable perspective.

Constantly, our some 75 main technical committees, on which are serving upwards of 8000 leaders in materials technology, are starting new research work and standards projects. Brief mention of some of these will give a concept of the diversity. A relatively new research tool, the electron microscope, is the subject of new standards covering sample preparation, etching, and replication. The Technical Committee on Paper is studying exhaustively different tests for flammability, to coordinate these. The Committee on Plastics completed an exhaustive discussion of working stresses for plastics. The new Carbon Black Committee is issuing a number of new methods, the first in this field. The Absorption Spectroscopy group has agreed on significant definitions and symbols. The Committee on Non-Ferrous Metals, which has concluded nationally famous corrosion tests, some extending over 20 years, is starting an extensive new series to be set up at four different locations, country-wide.

The Directors' Annual Report can hardly review all of the important technical activities that are under way, a number of them newly instituted during the year. Those concerned with specific fields can conveniently refer to the annual reports of the technical committees which are furnished the members on request. Major developments in the work of technical committees are covered in the various issues of the ASTM BULLETIN throughout the year.

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> "Definitions of terms and the study and formulation of test methods and specifica-

tions covering industrial casein, *isolated soya protein and similar proteinaceous materials.*"

This committee was organized in June of 1956.

Committee C-9 on Concrete and Concrete Aggregates, believing that a reference to "concrete products" would avoid misunderstandings on its field of activity, requested and received approval to change its scope by the addition of the words in italics:

> ". . . .the development of methods of test for concrete *and concrete products* and for the constituent materials of concrete. . . ."

**Electrical Contacts:**

Usually the ASTM standards for materials do not embody recommended dimensional standards, but in many instances data on dimensional standards as developed by other organizations are incorporated in appendices or in other parts of the respective specifications. Frequently such information is of distinct service to those using the ASTM standards.

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts, is developing a proposed tentative giving suggested dimensional standards for projection welding electrical contacts, and the publication of this was approved, to be appended to specifications as they are developed, or otherwise separately as a recommended practice with the note that the information will be handled as an appendix when specifications are issued.

**Steel, and Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:**

Acting on a request from the officers of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys, the Board of Directors considered the growing overlap of work between Committees A-1 on Steel and A-10. For a number of years there has existed a Joint Coordinating Committee consisting of representatives of these two committees and certain other committees working in the metals fields. Furthermore, in one area there is a joint subcommittee functioning between A-1 and A-10 relating to stainless tubing specifications. The Steel Committee through its advisory group has considered this matter. The Directors plan to call a meeting of appropriate officials of the two committees together with representation from the Board, at which consideration will be given to this involved matter.

## *New or Suggested Activities*

During the year progress was made in developing certain new activities, and there were many discussions either at conferences or in other ways on suggested new work.

*Cermets:*

Several technical committees are interested in these materials involving a combination of ceramics and metals. Representatives of Committee A-10 on Iron-Chromium and Iron-Chromium-Nickel Alloys, Committees B-9 on Metal Powders, C-21 on Ceramic Whitewares, and the Joint Committee on Effect of Temperature discussed this area at an all-day meeting and felt that a new approach should be made. Therefore a study committee is being appointed from which it is hoped specific recommendations on how best to organize will be forthcoming.

*Sorptive Mineral Materials:*

This new committee, carrying the designation C-23, was organized last February with the following scope:

> "The definition of terms and the formulation of test methods covering sorptive mineral materials used for surface maintenance and safety and the stimulation of research to accomplish the foregoing purposes."

The preliminary personnel of the committee and information on projected activities is covered on page 35 of the April ASTM BULLETIN.

*Industrial Chemicals:*

Discussions have been under way for over two years on undertaking additional work especially concerning methods of testing in the field of so-called industrial chemicals. Already many phases of this very extensive field are covered in ASTM committees, for example, Engine Antifreezes, Aromatic Hydrocarbons, Plastics, Paints, certain Petroleum Products, etc. Based on the latest conference with numerous leading organizations represented, it is proposed that an over-all technical committee be organized to cover general chemical methods of testing non-metallic materials (Committee E-3 on Chemical Analysis of Metals is effectively operating), and that smaller study groups review the desirability of organizing product committees to cover relatively small segments not now the subject of standardization work either in ASTM committees or elsewhere. It is the general feeling that the development of recognized standard methods of testing especially would be of considerable service.

*Flexible Packaging Materials:*

Since discussions have brought out that in any work on flexible packaging materials it will be the materials aspect that probably will be emphasized rather than evaluating these materials as shipping containers, ASTM Committee D-10 on Shipping Containers feels that it should not undertake this work under its present scope. A conference is being called of the people concerned with standards on these materials, to determine how best to proceed.

*Sampling of Materials:*

While numerous technical committees have carried out work on sampling and there are recommendations concerning this important aspect in various ASTM standards, it seems that more emphasis should be given to the use of statistical techniques in sampling. Committee E-11 on Quality Control of Materials is vitally interested in this subject. It sponsored a Symposium on Bulk Sampling in 1952 and through the work of a subcommittee has issued certain recommendations.

Committee D-5 on Coal and Coke obviously is intensely concerned, and a series of papers in this field was issued in 1955. Earlier certain technical papers were issued as a small compilation.

At a conference in New York in April, 1957, with leading experts participating, there was agreement that further emphasis should be considered and Committee E-11 is studying suggestions. It seems quite apparent that American industry could effect considerable savings by extending the statistical theory more effectively to sampling. Recommendations on different courses of action are expected.

*Nuclear Problems:*

Active consideration and work is under way in many ASTM technical committees concerned with the impact on materials of nuclear science. General guidance in this field is effected through a special Administrative Committee on Nuclear Problems reporting directly to the Board of Directors. At a meeting in February in Philadelphia called by the Administrative Committee, there was surprising attendance from representatives of ASTM technical groups, and there ensued an excellent discussion and review of the concern of various materials fields with aspects of the development and use of nuclear energy. The minutes of this meeting that are being distributed to all officers of the some 80 ASTM technical committees provide thumbnail sketches of the interest of the respective committees.

One very important activity here is the correlation and publication of information and data. At the September West Coast Meeting, there was the first of a series of joint symposiums on radiation effect on materials, this being sponsored by the Atomic Industrial Forum and ASTM. Three sessions were held. Another symposium will be featured at the June, 1957, Meeting of the Society, by the same sponsors.

An extensive Symposium on Radioactive Isotopes is being published. Another significant symposium dealing with Non-Destructive Tests in the Nuclear Energy Field, comprising a three-day series of papers, was held in Chicago in April, with ASTM as a joint sponsor. Some 40 papers, recently released by the AEC, were presented; ASTM is responsible for their publication.

Through the activity of its members and committee officers, and the guidance of the Administrative Committee, the Society intends to keep its activities involving research and standards in this field abreast of industry's needs in so far as possible.

It is highly important to conserve the efforts of our technical people and coordination of effort is essential as interest pyramids in this field of nuclear energy.

### *District and Related Activities*

The ASTM district activities continued in many ways to aid in furthering the work of the Society. News of most of these diverse activities has appeared in the ASTM BULLETIN, and all local meetings have been covered.

It will be noted that 36 local meetings were held during the past year. Included in this number were six meetings held in areas not currently incorporated in districts. Three of these meetings, Atlanta, Birmingham, and Jacksonville, were held in connection with the formation of a new Southeast District. It is expected that this area (Florida, Alabama, Georgia, South Carolina, Mississippi, and part of Tennessee) will be organized into a new district in 1957. The Society continued its cooperative efforts at the district level by holding 19 joint meetings.

President R. A. Schatzel presented his district address, "Materials for Electric Power Transmission," on 16 occasions. He addressed an additional four meetings. Dr. Charles E. Reed, the 1956 Marburg Lecturer, spoke before the Northern California and Southern California Districts, discussing "Silicones."

The Philadelphia District sponsored an evening's entertainment during the Spring Meeting held this year in Philadelphia. For the 1957 Annual Meeting in Atlantic City, the Philadelphia District will also sponsor the Ladies' Enter-

| | | |
|---|---|---|
| *Annual Meeting:* | | |
| 1956 Annual Meeting | June 17–22, 1956 | Atlantic City, N. J. |
| *Committee Week and Spring Meeting:* | | |
| 1957 Committee Week and Spring Meeting | February 4–8, 1957 | Philadelphia, Pa. |
| *District or Special Local Meetings:* | | |
| St. Louis District (jointly with AWS and Engineers Club of St. Louis) | October 11, 1956 | St. Louis, Mo. |
| Chicago District | October 16, 1956 | Chicago, Ill. |
| New England District (jointly with Society for Non-Destructive Testing, ASM and NACE) | October 25, 1956 | Cambridge, Mass. |
| Pittsburgh District (jointly with Pittsburgh Nuclear Activities Council) | October 30, 1956 | Pittsburgh, Pa. |
| Madison, Wis. (jointly with Technical Club) | November 5, 1956 | Madison, Wis. |
| Detroit District (jointly with Detroit Purchasing Agents Association) | November 15, 1956 | Detroit, Mich. |
| New York District (jointly with ASTE and ASME) | November 16, 1956 | New York, N. Y. |
| New York District (jointly with ASM) | November 19, 1956 | Rome, N. Y. |
| Philadelphia District (jointly with ASM) | November 29, 1956 | Philadelphia, Pa. |
| Southern California District (jointly with ACS) | December 7, 1956 | Los Angeles, Calif. |
| Northern California District (jointly with ACS and Golden Gate Paint and Varnish Production Club) | December 10, 1956 | San Francisco, Calif. |
| Jacksonville, Florida (jointly with ASM, other societies invited) | December 10, 1956 | Jacksonville, Fla. |
| Atlanta, Georgia | December 13, 1956 | Atlanta, Ga. |
| Washington, D. C. District (jointly with North Carolina State and The Quality Concrete Conference) | December 14, 1956 | Raleigh, N. C. |
| Southwest District | January 21, 1957 | Houston, Tex. |
| St. Louis District (jointly with Missouri Society of Professional Engineers) | January 22, 1957 | St. Louis, Mo. |
| Philadelphia District | February 5, 1957 | Philadelphia, Pa. |
| Northern California District (jointly with ASM) | February 11, 1957 | San Francisco, Calif. |
| Southern California District | February 12, 1957 | Los Angeles, Calif. |
| Southwest District (jointly with AIChE and ACS) | February 15, 1957 | New Orleans, La. |
| Southwest District (jointly with AIEE branch groups) | February 18, 1957 | Houston, Tex. |
| Rice Institute | February 19, 1957 | Houston, Tex. |
| Southwest District (jointly with AIEE) | February 21, 1957 | Dallas, Tex. |
| Richland, Washington (jointly with ASM, ASME, AIChE, ACS, ISA, AIEE and ASCE) | February 26, 1957 | Richland, Wash. |
| University of Idaho | February 27, 1957 | Moscow, Idaho |
| New York District | March 14, 1957 | New York, N. Y. |
| Philadelphia District | March 21, 1957 | Bethlehem, Pa. |
| Chicago District | March 27, 1957 | Chicago, Ill. |
| Birmingham, Alabama | April 16, 1957 | Birmingham, Ala. |
| Pittsburgh District | April 24, 1957 | Pittsburgh, Pa. |
| Cleveland District | April 25, 1957 | Cleveland, Ohio |
| New York District | April 25, 1957 | New York, N. Y. |
| Western New York-Ontario District | April 29, 1957 | Niagara Falls, Ont., Canada |
| New England District (jointly with University of Rhode Island and Providence Engineering Society) | May 3, 1957 | Kingston, R. I. |

tainment Program and the entertainment following the Annual Dinner.

Some districts have initiated new approaches to district meetings. New England is continuing its program of holding spring meetings at one of the colleges or universities in its area. This year the meeting was held at the University of Rhode Island. Chicago sponsored an afternoon forum on the future outlook of ASTM. Student Award Programs, combined with President's Nights, were held in seven districts—Detroit, Pittsburgh, Philadelphia, Cleveland, Northern California, Southern California and the Southwest (3). During the Second Pacific Area Meeting held in Los Angeles, the Northern and Southern California District Councilors held a Joint Breakfast Meeting.

The Administrative Committee on District Activities will discuss an extension of the "District Philosophy" to industrial areas where the number of members and committee members is not sufficient to warrant the establishment of a full District Council.

## *National Meetings*

### 1956 Annual Meeting

The 59th Annual Meeting at Atlantic City, June 17–22, established a new record high for attendance of 2900, and again more than 650 technical committee meetings were held, sometimes 60 running simultaneously. An outstanding technical program and apparatus exhibit were high lights. An interesting photographic exhibit brought out many good photographs and illustrations in the field of metallography and microscopy. A condensed article on the meeting, but still comprising 30 pages, was the main feature of the July 1956 ASTM BULLETIN.

### Outstanding Second Pacific Area National Meeting

In some ways this was the most extensive national meeting ever held by the Society. More than 200 technical papers required 43 sessions at the Hotel Statler in Los Angeles throughout the week of September 16. The attendance of 1537 registered did not include about a thousand visitors to the meeting and the exhibit. The West Coast Exhibit included not only displays in the field of apparatus and testing equipment, but also special displays covering the Society's work and the photographic prints were moved *en masse* from the June meeting. Seven industry luncheons and a President's luncheon were other features with attendance ranging from 100 to over 400. Nationally prominent men spoke, and leading executives, particularly of West Coast companies or branches, were at the head tables. The meeting not only emphasized to the Far West the importance and great breadth of ASTM work, but as at any national gathering, committees held meetings and many of the technical papers which are being issued as Special Technical Publications were outstanding. Full details have been furnished the members and the availability of the symposiums and papers will be widely publicized.

### 1957 Spring Meeting and Committee Week

ASTM Committee Week held each Spring offers technical committees an opportunity to schedule coordinated meetings and thus conserve the time of their members who may be serving on other committees. There were 300 such meetings during 1957 Committee Week in Philadelphia at the Benjamin Franklin Hotel, February 4–8.

The Spring Meeting held during Committee Week featured a Symposium on Thermal Insulation. Total registration for the week was just over 1300. This varies depending on the technical committees which participate and frequently many of the major committees wish to meet at other times elsewhere.

### Future Meetings

Annual Meetings of the Society have been scheduled as follows: June 16–21, 1957—Atlantic City, N. J.; June 22–27, 1958—Boston, Mass.; June 21–26, 1959, June 26–July 1, 1960—Atlantic City, N.J.

Committee Week and Spring Meetings: February 9–14, 1958, St. Louis, Mo.; February 2–7, 1959, Pittsburgh, Pa.; February 1–5, 1960, Chicago, Ill.; January 29–February 3, 1961, Cincinnati, Ohio.

### Local Committees Help

No national meetings of any society can be very successful without the close cooperation and effective help of local committees on arrangements. Too much credit cannot be given the General Committee for the outstanding success of the September West Coast meeting. Its subcommittees planned well and executed plans efficiently on finance, information, technical program, ladies' entertainment, plant visits, etc. The personnel of the committee has been given in various issues of the BULLETIN and in the program at the meeting. The President and the Staff have individually thanked the over 100 men who gave unstintingly of their time and effort. One result of this meeting was the knitting together more closely of our West Coast members and especially those serving on Southern and Northern California District Councils, which groups were the nucleus for the General Committee.

*Special Note on Philadelphia:*

Ever since its organization, the Philadelphia District through its active Council has been a leader in district activities, but of equal importance it has graciously acted as the host for the Annual Meetings when held in Atlantic City. The Council has been responsible for the ladies' entertainment, annual meeting dinner, and other features of the meeting. Each year it has raised funds to help in the underwriting. Undoubtedly the best accolade for this group are the many compliments from those at the Annual Meeting.

*New England:*

A General Committee on Arrangements for the 1958 Annual Meeting in New England is being organized. This group plans to make this meeting outstanding in many ways.

## *Administrative Matters*

*Headquarters:*

The area around the National Headquarters buildings continues to change in complexion. The new Sheraton Hotel is just a few blocks away. The buildings of the Moore School of Art in the same block are being completed. Much of the change comes from the extensive work on new highways and particularly the development of Pennsylvania Boulevard replacing the old railroad tracks leading to the now demolished Broad Street Station Building.

As the Staff continues to expand, it will be necessary to do further remodeling in the Cherry Street Building purchased a few years ago and perhaps eventually to construct a new building to join the Race Street and Cherry Street buildings. There seems to be no let-up in the demand for the Society to undertake research and standards activities, and the distribution of publications continues to grow.

*Headquarters Staff:*

In 1956 Mr. A. L. Batik, a graduate of Brooklyn Polytechnic, and the University of Cincinnati and of Buffalo, joined our Staff to assist in developmental activities including district work and to supervise BULLETIN and Index advertising promotion.

During the year some changes were made in the titles of certain Staff members more nearly to reflect their staff responsibilities as follows: Mr. G. A. Wilson, Production Editor, ASTM Publications; Mr. S. F. Etris, Junior Assistant Technical Secretary; and Miss Virginia Burkhart, Assistant Editor, ASTM BULLETIN.

## *Membership*

On May 1, 1957, the membership of the Society totaled 8836, compared with a total of 8323 one year ago. Gains and losses in various classes of membership are shown in the following table:

| Class of Membership | Membership | | Losses | | | | Additions | | Total | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | May 1, 1956 | May 1, 1957 | Resignations | Dropped | Death | Transfer | Transfer | Election | Loss | Gain | Increase |
| Honorary | 22 | 25 | ... | ... | 1 | ... | 4 | ... | 1 | 4 | 3 |
| Perpetuity and Life | 13 | 13 | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| Sustaining | 375 | 382 | 5 | ... | ... | ... | 3 | 9 | 5 | 12 | 7 |
| Company, Firm, etc. | 1859 | 1967 | 51 | 14 | ... | 6 | 13 | 166 | 71 | 179 | 108 |
| Individual, etc | 5919 | 6263 | 223 | 120 | 36 | 17 | 15 | 725 | 396 | 740 | 344 |
| Associate | 135 | 186 | 12 | 5 | 1 | 12 | ... | 81 | 30 | 81 | 51 |
| Total | 8323 | 8836 | 291 | 139 | 38 | 35 | 35 | 981 | 503 | 1016 | 513 |
| Student | 530 | 806 | 36 | 244 | ... | 15 | ... | 571 | 295 | 571 | 276 |

It is interesting to compare our present membership of 8836 with the figures for ten and twenty years ago; in 1947 our total membership was 6313, and in 1937 it was 3992.

Concerning the calendar year 1956, the number of new members was higher than any previous year, totaling 1014. The previous nine years ranged from 602 to 775. The net gain of 559 was higher than in any previous year.

The number of company and sustaining members continues to grow.

*Associate Membership:*

It is too early to assess the significance of a major change in our structure during the past year and a half, namely, dropping "Junior" membership and creating the new "Associate" class which extends to 30 years of age. Considerable promotional work has been done, and will continue, to make this more widely known, particularly in the engineering faculties. We are having good cooperation from professors in many of the leading schools. One of our objectives here was to attract younger engineering faculty members.

*Sustaining Members:*

There have been 12 new sustaining members in the period from May, 1956, to May, 1957, and five resignations. The new sustaining members are:

Canada Wire and Cable Co., Ltd.
Carbide and Carbon Chemicals Co.
Cast Iron Pipe Research Assn.
Eastern States Petroleum Co., Inc.
Evans Products Co.
Ford Motor Company of Canada, Ltd.
King & Gavaris
Litton Industries
Midwest Job Galvanizers Assn.
Oak Ridge National Laboratory
Polymer Chemicals Div., W. R. Grace and Co.
Silicones Div., Union Carbide and Carbon Corp.

*50-Year Members:*

There are four individuals and three organizations as noted below that have been connected with the Society continuously for 50 years, having joined in 1907:

Herbert Abraham
Henry C. Boynton
John Fairfield Thompson
Morton Owen Withey

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Reading Co.
Rensselaer Polytechnic Inst.
The Youngstown Sheet and Tube Co.

These members will be recognized at the Annual Meeting.

*40-Year Members:*

There are 31 members who this year have completed 40 years of membership, bringing the total number of certificates issued to the "Forty-Year Club" to 383. The members who will receive the 40-Year Certificates at this Annual Meeting are as follows:

Aluminum Company of America
American Cast Iron Pipe Co.
The Broken Hill Associated Smelters, Proprietary, Ltd.
Canadian Locomotive Co., Ltd.
Central Scientific Co.
The Chapman Valve Manufacturing Co.
University of Chile, Laboratory for Testing Materials
D-X Sunray Oil Co.
University of Delaware, Department of Mechanics
Dominion Foundries and Steel, Ltd.
General Petroleum Corp.
S. C. Hollister
Inland Steel Co.
Iowa State Highway Commission
A. H. Jameson
City of Los Angeles, Bureau of Standards
Marblehead Lime Co.
Lawrence S. Marsh
Mesta Machine Co.
The National Cash Register Co.
National Lumber Manufacturers Assn.
K. T. Potthoff
City of St. Paul, Bureau of Municipal Testing Labs.
The Henry Souther Engineering Co.
Stewarts & Lloyds, Ltd.
E. Winthrop Taylor
Richard L. Templin
Townsend Co.
United States Smelting, Refining, and Mining Co.
Vanadium Corporation of America
Vulcan Materials Co.

*Deaths:*

The Society has lost the following 38 members through death (figures in parentheses are dates of membership):

Anderegg, F. O. (1929)
Beckstrand, E. H. (1907)
Berrigan, Thomas A. (1946)
Bissell, Clinton T. (1929)
Boardman, Harry C. (1949)
Breth, F. W. (1923)

Broad, J. L. (1953)
Camp, William M. (1939)
Christie, Alexander J. (1907)
Churchill, Harry D. (1943)
Cohen, A. Burton (1913)
Connor, James E. (1950)
Daly, Benedict T. (1955)
Dill, Richard S. (1948)
Emerson, Charles J. (1920)
Fulton, A. O. (1925)
Hallenbeck, G. S. (1928)
Harder, Oscar E. (1920)
Hardesty, Shortridge (1938)
Holmes, H. H. (1948)
Jacobs, Nathan B. (1937)
Kawin, Charles C. (1936)
Kempka, Gilbert E. (1955)
Lever, Harry R. (1956)
Maag, O. L. (1936)
Macelwane, James B. (1948)
Magnel, Gustave (1950)
McMullen, E. W. (1945)
Morze, Eugene W. (1952)
Murdoch, B. M. (1944)
Pearse, Langdon (1923)
Powell, E. N. (1952)
Saathoff, George W. (1924)
Schlissel, M. M. (1934)
Storer-Felt, John P. (1956)
Whitcomb, William H. (1926)
*White, Albert E. (1914)
Wolf, Harry R. (1913)

In addition to the above, the following representatives of company members of the Society passed away:

L. L. Adams, Louisville & Nashville Railroad Co.
S. W. Andrews, H. G. Acres and Co., Ltd.
S. A. Brazier, Dunlop Rubber Co., Ltd.
E. O. Dixon, Ladish Co.
Alexander Frieden, Pabst Laboratories
Howard F. Kichline, North American Cement Co.
Frank P. Leahey, Nichols Wire and Aluminum Co.
Frank W. Lewis, Chicago Railway Equipment Co.
Charles Nowell, Manchester Public Library
Jonathan Sharp, Minneapolis Honeywell Regulator Co., Brown Instruments Div.
H. A. Smith, Corporation of the City of Durban
T. B. Taylor, American Creosoting Co.
Howard McCutcheon Thomas, Fort Scott Hydraulic Cement Co.

A number of these men have been affiliated with the Society for many years and rendered outstanding services, particularly through work on the technical committees. The various issues of the ASTM BULLETIN have included appropriate notices and appreciation of their work.

## *Honors and Awards*

Last year it had been agreed to allow two years to elapse between the presentation of technical papers and the conferring of awards therefor. The transition period now having expired, awards for outstanding papers are once again on an annual basis and will be presented at the coming annual meeting. Descriptions of the various awards as well as the personnel of the lecture and award committees are given in the ASTM Year Book.

Recipients of honors and awards to be given at the 1957 Annual Meeting are indicated below:

*Award of Merit:*

J. W. Bolton
D. L. Colwell
A. G. H. Dietz
T. P. Dresser, Jr.
P. V. Faragher
E. W. Fasig
W. A. Kennedy
W. H. Koch
R. E. Penrod
F. W. Reinhart
F. G. Tatnall
W. C. Voss

*Recognition of Technical Papers:*

*Charles B. Dudley Medal* (research).—R. U. Blaser and J. J. Owens for their paper "Special Corrosion Study of Carbon and Low-Alloy Steels."

*Richard L. Templin Award* (testing).—W. Ramberg and L. K. Irwin for their paper "Longitudinal Impact Tests of Long Bars with a Slingshot Machine."

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* Honorary Member.

*Sam Tour Award* (corrosion).—K. G. Compton and A. Mendizza for their paper "Galvanic Couple Corrosion Studies by Means of the Threaded Bolt and Nut Wire Test."

*Sanford E. Thompson Award* (concrete, sponsored by Committee C-9).—T. C. Powers for his paper "Basic Considerations Pertaining to Freezing-and-Thawing Tests."

The system of rating the presentations of technical papers has been continued, and the persons receiving the three highest ratings for outstanding presentations will be given recognition by a letter from the President of the Society and an announcement in the ASTM BULLETIN.

*Recognition of Outstanding Contributions:*

*Frank E. Richart Award* (concrete).—Stanton Walker, Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

*Harold DeWitt Smith Memorial Medal* (textiles).—William D. Appel, Assistant Chief, Organic and Fibrous Materials Div., National Bureau of Standards, Washington, D. C.

*Max Hecht Award* (industrial water, sponsored by Committee D-19).—Robert C. Adams, Superintendent, Chemical Engineering Lab., U. S. Naval Engineering Experiment Station, Annapolis, Md.

*ASTM Lectures:*

The *Edgar Marburg Lecture* on the subject "Your Most Important Raw Material" (industrial water) will be presented on Wednesday afternoon, June 19, at Haddon Hall, by Everett P. Partridge, Director, Hall Laboratories, Inc., Pittsburgh, Pa. The *H. W. Gillett Memorial Lecture* on the subject "A Perspective of Molybdenum-Base Alloys," will be presented on Tuesday afternoon, June 18, also at Haddon Hall, by Alvin J. Herzig, President, Climax Molybdenum Co. of Michigan, Detroit, Mich.

## *Finances*

*Report for 1956:*

Full details of the fiscal operations of the Society for 1956 are included in the report of the auditors appearing in Appendix I.[1]

*Operating Receipts:*

Our expectations that 1956 would be a good year income-wise were more than realized. The estimated receipts of $967,500 actually developed to be $1,082,522. Of this amount, receipts from dues and entrance fees were $268,985 or 24.8 per cent; receipts from sales of publications were $695,950 or 64.3 per cent; and receipts from miscellaneous sources consisting principally of advertising, income from investments, exhibits and registration fees totaled $117,617 or 10.9 per cent.

It will be noted that receipts from membership fees are again, for the fourth consecutive year, under 30 per cent of the total.

*Operating Disbursements:*

Total disbursements were $896,514, not including $85,000 earmarked for the 1958 Book of Standards. For this book, we build up a reserve over a three year period.

A total of $342,323 was spent for publications, which compares with $349,962 for the previous year. Expenditures for salaries, which included extra summer help and considerable overtime, reached $309,400. This figure represents 34.3 per cent of total disbursements. Cost of employee benefits, including the major medical policy and covering new participants in the retirement plan, totaled $32,603.

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[1] This report is not included in the preprint but will be published in the 1957 *Proceedings*. A limited number of proofs are available and will be sent on request, or can be procured at the Annual Meeting.

*Balance Sheet:*

On November 30 our total assets in General Funds were $819,600 compared to $736,154 a year ago. In all funds—General, Building, Medal, etc.—total assets were $1,473,516 compared with $1,344,743 a year ago. It should be pointed out that in the Balance Sheet no account has been taken of the assets of the Society in the form of publications in stock. This is consistent with many years' practice in this respect and, of course, results in a more conservative statement. An inventory of the principal technical publications as of May 1, 1957, is summarized below:

| | |
|---|---|
| Proceedings (All back copies from 1955) | 2 517 |
| 1955 Book of ASTM Standards | |
| Part 1 | 2 319 |
| Part 2 | 2 163 |
| Part 3 | 2 070 |
| Part 4 | 1 566 |
| Part 5 | 1 884 |
| Part 6 | 1 466 |
| Part 7 | 1 850 |
| 1956 Supplement to Book of ASTM Standards | |
| Part 1 | 6 015 |
| Part 2 | 3 884 |
| Part 3 | 3 416 |
| Part 4 | 2 742 |
| Part 5 | 2 598 |
| Part 6 | 2 690 |
| Part 7 | 2 370 |
| Special Compilation of Standards | 15 590 |
| Selected ASTM Engineering Materials Standards | 8 994 |
| Symposiums and Special Reprints | **147 169** |
| Reprints of Standards (Approx.) | **240 000** |
| Radiographic Standards for Steel Castings | 24 |
| Radiographic Standards for Aluminum & Magnesium Castings | 30 |

*Not included in inventory:*

(1) Viscosity Temperature Charts
(2) Hardenability Charts
(3) Manual of Engine Test Methods and Supplement
(4) Manual for Rating Motor Fuel
(5) Marburg Lectures
(6) Cotton Yarn Boards
(7) Petroleum Charts

*Building Fund:*

Assets in the Building Fund, which include the current Race Street Headquarters, the Cherry Street property, and the balance of the lot immediately adjoining this, total $307,052.

The loan from ASTM General Funds which enabled the procurement of the Cherry Street property now amounts to $46,725 as of November 30, 1956. It is being amortized in the amount of $8700 yearly and should be wiped out accordingly in about 5 years.

*Investments:*

In the operation of the Investment Fund, the Society had continuing advice from our investment counsel, Loomis, Sayles & Co. This Fund was set up in 1956 by transferring to it assets from the various operating funds such as General, Building, Research, etc., with these funds owning *pro rata* a share in the Investment Fund. The operating funds proportionately benefit from income and growth of the Fund. In January, 1957, the operating funds transferred further assets in the amount of $75,000.

During the year a number of changes in investments were made, some bonds and stocks being sold and a number of additional securities purchased. These are detailed in the Auditors' Report which is appended.[1]

As of November 30, the book value or cost of the securities in the fund totaled about $708,000; market value was $871,700. Total income was $33,750.

In recent months purchases have been made approximately in the ratio of holdings which prevailed in the fund in late 1956, namely, about 46 per cent bonds, preferreds and cash; 54 per cent common stocks. The Society's holdings of mutual funds are included in the totals of the Investment Fund. As of November 30 mutual holdings had a market value of about $207,500.

*1957 Budget:*

From the accompanying summary of the 1957 budget, it will be seen that a deficit is anticipated for the current year. This is a relatively normal expectation because our operations really are on a three-year basis, influenced by the triennial Book of Standards, and the income from the 1955 Book will fall off sharply during this year whereas it was at a peak in 1956, thus creating the very favorable balance in that year. Such funds as are necessary to make up the difference between income and disbursements will be drawn from the Society's surplus.

1957 BUDGET SUMMARY

Estimated Receipts

| | | Per Cent |
|---|---|---|
| Dues and Entrance Fees | $275 000 | 29.1 |
| Publications | 574 500 | 60.9 |
| Miscellaneous | 93 500 | 10.0 |
| TOTAL CURRENT RECEIPTS | $943 000 | |
| Publications | $406 500 | 40.0 |
| Salaries | 380 500 | 37.3 |
| General Office Expenses | 93 500 | 9.2 |
| Technical Committee Expenses | 11 800 | 1.2 |
| Meetings | 16 000 | 1.5 |
| Headquarters Occupancy Expenses | 42 500 | 4.2 |
| Retirement, Health | 44 500 | 4.4 |
| Miscellaneous Expenses | 22 100 | 2.2 |
| TOTAL OPERATING DISBURSEMENTS | $1 017 400 | |

## *Cooperative Activities Essential in Materials Research and Standardization*

One might consider our extensive cooperative contacts with many other societies and trade associations as but another aspect of our basic premise in bringing together on technical committees the representatives of the consumer and the producer. In essence the situation is parallel, but cooperation among various societies concerned with materials is so essential in avoiding duplication of effort that special reference to some of our cooperative work is justified.

In ASTM there are upwards of 250 professional and technical societies, institutes, and trade associations which hold membership and which in most cases are vitally concerned with our work in certain technical areas. Many representatives of these groups take a leading part in the various technical committees.

With engineering and scientific manpower in heavy demand, obviously an organization such as ASTM cutting horizontally across the materials field must use available manpower as efficiently as possible. To this end, we not only welcome but urge the participation in our work of other organizations. The record is an inspiring one.

Officers of the Society and of our technical committees, as well as the Staff, constantly are discussing our work with officials of other organizations and their committees. There are an increasing number of jointly sponsored committees, particularly in relatively newer areas such as reinforced plastic and plastic pipe, cellulose, etc.

A number of the joint activities go back many years. For example, Technical Committee "A" on Automotive Rubber is a joint project of the SAE and ASTM; and the Joint Committee on Effect of Temperature is jointly sponsored by the ASME and ASTM.

During the year important contacts have been made with many groups, and agreements of cooperation have been established with a number of these. Problems of a diverse nature involving facets of both research and standardization have been discussed, for example, with the following organizations:

Atomic Industrial Forum
Aircraft Industries Association
American Association of Textile Chemists and Colorists
American Chemical Society
American Institute of Electrical Engineers
American Society of Mechanical Engineers
American Society of Tool Engineers
Manufacturing Chemists' Association
National Association of Corrosion Engineers
Chemical Specialties Manufacturers Association
Technical Association of the Pulp and Paper Industry
Society of Plastics Engineers

*Government Contacts:*

Pursuing the policy of cooperating with the Federal Government in avoiding duplication of standards and attempting better to coordinate industry and Government standards, additional conferences have been held with the General Services Administration responsible for Federal Specifications work, and the Department of Defense, specifically the officials responsible for Cataloging, Inspection, and Standardization. There are several outstanding examples of effective coordination of industry and Government Standards. It is hoped there will be more and closer ties between ASTM and the Government divisions responsible for groups of standards.

The Society has been following rather closely the development, under the National Research Council, of the Building Research Advisory Board and the Building Research Institute.

Our good relations continue with numerous purchasing agent organizations, such as the National Association of Purchasing Agents, the National Institute of Governmental Purchasing, and the National Association of State Purchasing Officials. This latter group has been conducting an extensive survey on the use of standards.

*Engineering Education:*

The Society is vitally concerned with the future supply of engineers and scientists. During the year many contacts have been made with leading universities and schools in connection with ASTM student membership prize awards plan, visits by the President and the Executive Secretary, and in other ways. The Society's future ties in with this matter of the education of our youth.

When we consider where we are going, the President in his West Coast Luncheon address[2] partially at least answers this question by indicating there is little doubt that ". . . we go forward to accept the challenge of obtaining better materials and performance. . . . The rate at which we accomplish this will depend on the continued coordination of technical knowledge through organizations such as ASTM and the character and quality of education of our youth."

Respectfully submitted on behalf of the Board of Directors,

R. A. SCHATZEL,
*President.*

R. J. PAINTER,
*Executive Secretary.*

June, 1957

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[2] ASTM BULLETIN, No. 217, Oct., 1956, p. 21.

## APPENDIX I

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## REPORT OF THE AUDITORS FOR THE FISCAL YEAR DECEMBER 11, 1955 TO NOVEMBER 30, 1956

Philadelphia, December 19, 1956

MR. R. J. PAINTER, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the period December 11, 1955 to November 30, 1956. We did not make a detailed audit of all transactions, but made tests to the extent we considered appropriate in determining the accuracy of the accounts.

Cash on deposit in checking and savings accounts was verified by direct correspondence with the various depositories and reconciled with the cash records. Accounts receivable were not verified by correspondence with debtors. The securities held by the investment fund and certificates of deposit were examined by us on December 19, 1956. Income from investments for the period under review was verified by us and found to be properly recorded on the books of the society.

We have prepared and submit herewith balance sheet as of November 30, 1956, and statement of cash receipts and disbursements and operations for the period December 11, 1955 to November 30, 1956, and other supporting schedules which, in our opinion, present fairly the financial position of the society at November 30, 1956, and the results of its operations for the period ended that date.

Respectfully submitted,
JOHN HEINS & CO.

## BALANCE SHEET AS OF NOVEMBER 30, 1956

(Including Special and Designated Funds)

### ASSETS

| | | | | |
|---|---|---|---|---|
| *General Funds:* | | | | |
| Current: | | | | |
| Cash | | $194 447.80 | | |
| Less Check No. 2073 as of November 30, 1956 (drawn against estimated cost of 1956 *Proceedings* and 1956 Supplement to 1955 Book of Standards) | | 68 000.00 | $126 447.80 | |
| Investment Fund Participation (at cost) | | | 537 388.15 | |
| Accounts Receivable | | | 75 619.22 | |
| Loans Receivable (Building Fund) | | | 46 725.00 | |
| Advance to Forest Products Laboratory | | | 500.00 | |
| Total Current Assets—General Funds | | | 786 680.17 | |
| Furniture and Fixtures (depreciated book value) | | | 32 909.04 | |
| Total Assets—General Fund | | | | $819 589.21 |
| *Building Fund:* | | | | |
| Cash | | | 16 646.75 | |
| Investments Fund Participation (at cost) | | | 20 961.02 | |
| Land—Cost | | | 68 459.42 | |
| Building and construction costs | | 264 785.41 | | |
| Less, accumulated depreciation | | 63 800.00 | 200 985.41 | |
| Total Assets—Building Fund | | | | 307 052.60 |
| *Other Special and Designated Funds:* | | | | |
| Cash: | | | | |
| ASTM Research Fund | | 15 612.44 | | |
| Dudley Medal and Marburg Lecture Fund | 705.13 | | | |
| Richard L. Templin Award Fund | 172.99 | | | |
| Sam Tour Award Fund | 309.92 | | | |
| F. E. Richart Award Fund | 91.23 | | | |
| Total Medal and Lecture Funds | | 1 279.27 | | |
| Committee Funds | | 103 925.50 | 120 817.21 | |
| *Investments Fund Participations (at cost):* | | | | |
| ASTM Research Fund | | 67 337.28 | | |
| Dudley Medal and Marburg Lecture Fund | | 6 943.33 | | |
| Richard L. Templin Award Fund | | 2 221.87 | | |
| Sam Tour Award Fund | | 1 729.28 | | |
| F. E. Richart Award Fund | | 1 048.05 | | |
| Committee Funds | | 100 193.67 | 179 473.48 | |
| *Other Investments (at cost):* | | | | |
| ASTM Research Fund | | 7 258.63 | | |
| Committee Funds | | 39 325.00 | 46 583.63 | |
| Total Assets, Special and Designated Funds | | | | 346 874.32 |
| Total Assets | | | | $1 473 516.13 |

## LIABILITIES

| | | | |
|---|---|---|---|
| *General Funds:* | | | |
| Current: | | | |
| Advance Collections | | $19 712.33 | |
| Accounts Payable: | | | |
| Committee C-1 | | 146.63 | |
| Joint Committee on X-ray Diffraction | | 130 190.65 | |
| Institute of Petroleum | | 3 873.46 | |
| Pennsylvania Sales Tax | | 97.48 | |
| Total Current Liabilities—General Funds | | 154 020.55 | |
| Life Membership Fund | $4 017.94 | | |
| Publication Fund | 16 841.80 | | |
| Book of Standards Reserve | 86 030.02 | | |
| Executive Retirement Reserve | 17 173.60 | | |
| Retirement Fund Reserve | 36 398.89 | | |
| Reserve for Depreciation of Investments | 20 000.00 | | |
| Reserve for Additional Cost of Replacement of Headquarters Building | 35 000.00 | | |
| Special Annual Meeting Entertainment Account | 5 586.64 | | |
| | 221 048.89 | | |
| Surplus | 444 519.77 | 665 568.66 | |
| Total Liabilities and Surplus—General Funds | | | $819 589.21 |
| *Building Fund:* | | | |
| Contributions from Members | | 181 054.42 | |
| Contributions from ASTM General Funds | | 73 364.57 | |
| Notes Payable—General Funds | | 46 725.00 | |
| Accumulated Income, net | | 5 908.61 | |
| Total Building Fund | | | 307 052.60 |
| *Other Special and Designated Funds:* | | | |
| ASTM Research Fund: | | | |
| Principal | 81 851.28 | | |
| Income | 8 357.07 | 90 208.35 | |
| Dudley Medal and Marburg Lecture Fund: | | | |
| Principal | 6 943.33 | | |
| Income | 705.13 | 7 648.46 | |
| Richard L. Templin Award Fund: | | | |
| Principal | 2 221.87 | | |
| Income | 172.99 | 2 394.86 | |
| Sam Tour Award Fund: | | | |
| Principal | 1 729.28 | | |
| Income | 309.92 | 2 039.20 | |
| F. E. Richart Award Fund: | | | |
| Principal | 1 048.05 | | |
| Income | 91.23 | 1 139.28 | |
| Committee Funds, Unexpended Balance | | 243 444.17 | |
| Total Special and Designated Funds | | | 346 874.32 |
| Total Liabilities and Funds | | | $1 473 516.13 |

## In Addition to the General Funds of the Society the Treasurer had on Hand the Following Funds as of November 30, 1956

### COMMITTEE FUNDS

| | |
|---|---|
| Committee A-1 On Steel | 3 894.17 |
| Committee A-5 On Corrosion of Iron and Steel | 466.84 |
| Committee B-3 On Corrosion of Non-Ferrous Metals and Alloys | 2 913.01 |
| Committee B-5 Weaver Award Fund | 17.00 |
| Committee B-6 On Die-Cast Metals and Alloys | 3 161.62 |
| Committee B-8 On Electrodeposited Metallic Coatings | 11.25 |
| Committee C-1, On Cementitous, Ceramic, Concrete and Masonry—General Funds | 1 839.88 |
| Committee C-1 Cement Reference Laboratory | 831.13 |
| Committee C-9 Sanford E. Thompson Medal Fund | 1 142.81 |
| Committee C-9 On Concrete and Concrete Aggregates | 231.77 |
| Committee C-15 On Manufactured Masonry Units | 521.55 |
| Committee C-18 On Natural Building Stones | 426.83 |
| Committee D-1 On Paint, Varnish, Lacquer and Related Products | 795.32 |
| Committee D-2 On Petroleum Products and Lubricants | 1 267.09 |
| Committee D-2 ASTM-IBM | 1 053.41 |
| Committee D-2 D.C.C Reference Fuel Account | 33 548.60 |
| Committee D-2 D.C.C National Exchange Group | (2 298.23) |
| Committee D-4 On Road and Paving Materials | 987.53 |
| Committee D-5 On Coal and Coke | 819.58 |
| Committee D-7 On Wood | 44.50 |
| Committee D-7 Wood Pole Research Fund | 25 139.40 |
| Committee D-9 On Electrical Insulating Materials | 2 780.75 |
| Committee D-10 On Shipping Containers | 75.89 |
| Committee D-11 On Rubber—Special | 578.66 |
| Committee D-11 On Rubber and Rubber-like Materials | 358.97 |
| Committee D-12 On Soaps and Detergents | 1 040.15 |
| Committee D-13 On Textile Materials | 1 103.17 |
| Committee D-14 On Adhesives | 66.41 |
| Committee D-15 On Engine Antifreezes | 43.00 |
| Committee D-18—C. A. Hogentogler Award Fund—Soil Testing Procedures | 16.26 |
| Committee D-19—Max Hecht Award Fund—Industrial Water | 68.56 |
| Committee D-20 On Plastics | 102.00 |
| Committee E-2 On Emission Spectroscopy Analysis | 81.14 |
| Committee E-3 On Chemical Analysis of Metals | 198.39 |
| Committee E-4 On Metallography | 65.00 |
| Committee E-9 On Fatigue of Metals | 279.12 |
| Committee E-14 On Mass Spectrometry | 2 417.49 |
| ASA Sectional Committee on Specifications for Cast Iron Pipe | 1 546.20 |
| Chicago District Council | 3 680.40 |
| Cleveland District | 518.05 |
| Detroit District | 3 008.65 |
| Ohio Valley District | 4.50 |
| Philadelphia District | 669.52 |
| Philadelphia District Special (Annual Meeting Account) | 1 978.93 |
| Southern California District | 3 199.81 |
| Northern California District | 2 000.00 |
| Reserve-Future-West Coast Meetings | 7 000.00 |
| Pittsburgh District | 355.75 |
| Washington District | 16.23 |
| Western New York—Ontario District | 1 926.46 |
| Advisory Committee on Corrosion | 58 695.82 |
| Administrative Committee on Ultimate Consumer Goods | 283.11 |
| Joint Committee on X-ray Diffraction, chemical analysis | 64 754.33 |
| Joint Committee on X-ray Diffraction, research fellowship | 7 616.39 |
| Joint Committee on X-ray Diffraction, IBM calculator | 100.00 |
| | 444.17 |

Accounted for as follows:

| | | | |
|---|---|---|---|
| Cash in bank and on hand | | $103 925.50 | |
| Investments (at cost)—(market value $37 706.25) | | | |
| $20 000 U. S. Treasury 2½s 8/15/63 | $19 925.00 | | |
| $20 000 U. S. Treasury 2½s 11/15/61 | 19 400.00 | 39 325.00 | |
| Investment Fund Participation | | 100 193.67 | $243 444.17 |

### DUDLEY MEDAL AND MARBURG LECTURE FUND

#### *Principal Account*

| | *Total* | *Invested* |
|---|---|---|
| Balance, December 10, 1955 | $6 706.25 | $6 706.25 |
| Receipts: Transfer from Income (Stock Dividends) | 55.42 | 55.42 |
| Transfer from Income (Gain on Sale of Investments) | 181.66 | 181.66 |
| Balance, November 30, 1956 | $6 943.33 | $6 943.33 |

#### *Income Account*

| | | |
|---|---|---|
| Balance, December 10, 1955, as reported | $819.77 | |
| Receipts, Interest, Dividends and Profit on Investments | 537.89 | $1 357.66 |
| Disbursements: Honorarium | 400.00 | |
| Miscellaneous | 15.45 | |
| Transfer to Principal Account | 237.08 | 652.53 |
| Balance, November 30, 1956 | | $705.13 |

### RICHARD L. TEMPLIN AWARD FUND

#### *Principal Account*

| | | | *Uninvested Cash* |
|---|---|---|---|
| Balance, December 10, 1955, as reported | $2 146.00 | $2 146.00 | |
| Receipts: Transfer from Income (Stock Dividend) | 17.74 | 17.74 | |
| Transfer from Income (Gain on Sale of Investments) | 58.13 | 58.13 | |
| Balance, November 30, 1956 | $2 221.87 | $2 221.87 | 0 |

#### *Income Account*

| | | |
|---|---|---|
| Balance, December 10, 1955, as reported | $76.74 | |
| Receipts: Interest: Dividends and Profit on Investments | 172.12 | $248.86 |
| Disbursements: Transfer to Principal Account | | 75.87 |
| Balance, November 30, 1956 | | $172.99 |

### SAM TOUR AWARD FUND

#### *Principal Account*

| | | |
|---|---|---|
| Balance, December 10, 1955, as reported | $1 670.24 | $1 670.24 |
| Receipts: Transfer from Income Account (Stock Dividends) | 13.80 | 13.80 |
| Transfer from Income (Gain on Sales of Investments) | 45.24 | 45.24 |
| Balance, November 30, 1956 | $1 729.28 | $1 729.28 |

#### *Income Account*

| | | |
|---|---|---|
| Balance, December 10, 1955, as reported | $235.01 | |
| Receipts: Interest, Dividends and Profit on Investments | 133.95 | $368.96 |
| Disbursements: Transfer to Principal Account | | 59.04 |
| Balance, November 30, 1956 | | $309.92 |

## ASTM RESEARCH FUND

### *Principal Account*

| | *Total* | *Invested* | *Uninvested Cash* |
|---|---|---|---|
| Balance, December 10, 1955, as reported | $74 996.60 | $72 296.60 | $2 700.00 |
| Receipts: Stock Dividends Transferred from Income | 537.53 | 537.53 | |
| Profit on Sales of Investments | 1 761.78 | 1 761.78 | |
| Transferred from General Funds | 3 200.00 | | 3 200.00 |
| Final Distribution—H. J. Love Estate | 1 355.37 | | 1 355.37 |
| Balance, November 30, 1956 | $81 851.28 | 74 595.91 | $7 255.37 |
| Investment Fund Participation | $67 337.28 | | |
| 618 Sh General Motors Co. Common (Book Value)[a] (market value $27 037.50) | 7 258.63 | $74 595.91 | |

### *Income Account*

| | | |
|---|---|---|
| Balance, December 10, 1955, as reported | $5 502.31 | |
| Receipts: Interest, Dividends and Profit on Investments | 5 216.57 | $10 718.88 |
| Disbursements: Transfers to Principal Account | 2 299.31 | |
| Contribution to Committee E-14 | 62.50 | 2 361.81 |
| Balance, November 30, 1956 | | $8 357.07 |

## F. E. RICHART AWARD FUND

### *Principal Account*

| | | |
|---|---|---|
| Balance, December 10, 1955 | $1 012.26 | 1 012.26 |
| Receipts: Transfer from Income Account—Stock Dividend | 8.37 | 8.37 |
| Transfer from Income Account—Gain on Sale of Investments | 27.42 | 27.42 |
| Balance, November 30, 1956 | $1 048.05 | $1 048.05 |

### *Income Account*

| | | |
|---|---|---|
| Balance, December 10, 1955 | $45.82 | |
| Receipts: Interest, Dividends and Profit on Investments | 81.20 | $127.02 |
| Disbursements, Transfer to Principal Account | | 35.79 |
| Balance, November 30, 1956 | | $91.23 |

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Income from these shares is by agreement paid to an annuitant.

## RECEIPTS AND DISBURSEMENTS

### FOR THE PERIOD DECEMBER 11, 1955 TO NOVEMBER 30, 1956

| | | | |
|---|---|---|---|
| Cash balance, December 10, 1955 | | | $30 625.00 |

### RECEIPTS

| | | | |
|---|---|---|---|
| *Operating Receipts (Budgeted):* | | | |
| *Dues and Entrance Fees:* | | | |
| Current Dues | $257 951.66 | | |
| Life membership | 175.00 | | |
| Past Dues | 991.25 | | |
| Advance Dues | 3 391.74 | | |
| Entrance Fees | 6 475.60 | | |
| Total Dues | | $268 985.25 | |
| *Sales of Publications:* | | | |
| Book of Standards (Members, additional parts) | 58 238.35 | | |
| 1952 Book of Standards | 3 067.05 | | |
| 1955 Book of Standards | 312 975.62 | | |
| 1953 and 1954 Supplements to Book of Standards | 3 824.60 | | |
| 1956 Supplement to Book of Standards | 2 541.45 | | |
| Methods of Chemical Analysis of Metals | 2 423.15 | | |
| Compilation of Standards | 99 597.45 | | |
| Separate Standards | 43 743.39 | | |
| Selected Standards for Students | 5 073.80 | | |
| Proceedings | 8 639.65 | | |
| Subscriptions (nonmembers) | 3 931.11 | | |
| Special and Miscellaneous Publications | 151 894.36 | | |
| Total Sales of Publications | | 695 949.98 | |
| *Miscellaneous:* | | | |
| Advertising—BULLETIN | 40 254.80 | | |
| Advertising—Index | 3 069.51 | | |
| Interest and Dividends | 29 797.64 | | |
| Registration and Other Fees—Meetings | 16 622.45 | | |
| ASTM Exhibit at Annual Meeting | 20 168.50 | | |
| Staff Services | 6 995.63 | | |
| Miscellaneous | 708.59 | 117 617.12 | |
| Total Operating Receipts (Budgeted) | | 1 082 552.35 | |
| *Nonoperating Receipts (Not Budgeted):* | | | |
| Excess Remittance | 3 478.92 | | |
| Committee C-1 for Technical Assistant | 1 900.00 | | |
| Retirement Fund Reserve | 3 411.54 | | |
| Sale of X-ray Diffraction Cards | 116 902.97 | | |
| Viscosity Index | 1 183.27 | | |
| Institute of Petroleum Publications | 611.30 | | |
| Pacific Area Meeting to be Refunded | 1 371.79 | | |
| Buffet Supper | 299.00 | | |
| From Building Fund on Account of Loan | 7 975.00 | | |
| Annual Meeting Luncheon | 1 320.00 | | |
| Gain, Sale of Investments | 14 059.93 | | |
| Gillett Memorial | 564.90 | | |
| Repayment of Investment Fund Note | 55 000.00 | | |
| Miscellaneous | 10.83 | | |
| Total Nonoperating Receipts | | 208 089.45 | |
| Total Receipts | | | 1 290 641.80 |
| Total Receipts for 1956 and Cash Balance, December 10, 1955 | | | $1 321 266.80 |

## DISBURSEMENTS

| | | | |
|---|---|---|---|
| *Operating Disbursements (Budgeted):* | | | |
| *Publications:* | | | |
| Year Book | 13 075.92 | | |
| 1952 Book of Standards | 126.65 | | |
| 1955 Book of Standards | 19 021.41 | | |
| 1953 and 1954 Supplements to Book of Standards | (47.62)[b] | | |
| 1956 Supplement to Book of Standards | 50 375.72 | | |
| Methods of Chemical Analysis of Metals | 1 284.18 | | |
| Compilation of Standards | 37 741.60 | | |
| Separate Standards | 14 817.25 | | |
| Proceedings—repurchase of old numbers | 92.00 | | |
| 1955–1956 Proceedings | 41 176.75 | | |
| Preprints | 10 903.80 | | |
| ASTM BULLETIN | 45 426.51 | | |
| Circulars to members | 4 613.85 | | |
| Index to Standards | 10 475.02 | | |
| Special and Miscellaneous Publications | 93 229.30 | | |
| West Coast—Papers and Preprints | 10.76 | | |
| Total Disbursements—Publications | | $342 323.10 | |
| Salaries | 309 401.96 | | |
| General Office Expenses | 111 557.61 | | |
| Expenses—Technical and District Committees | 11 752.62 | | |
| Expenses—Meetings | 30 453.25 | | |
| Headquarters Occupancy Expense (includes $9 700 depreciation on building) | 35 581.98 | | |
| Transfer to Principal of Research Fund | 3 200.00 | | |
| American Standards Association | 1 500.00 | | |
| Traveling Expense, Administrative and Special Committees | 8 502.99 | | |
| Furniture and Fixtures | 8 127.99 | | |
| Dues, Contributions, Miscellaneous | 1 508.75 | | |
| Group Insurance | 813.15 | | |
| Employees' Retirement Fund | 23 530.49 | | |
| Federal Old Age Survivors | 4 886.73 | | |
| Blue Cross | 1 235.46 | | |
| Major Medical | 2 137.41 | 554 190.39 | |
| Total Operating Disbursements (Budgeted) | | 896 513.49 | |
| *Nonoperating Disbursements (Not Budgeted):* | | | |
| Refund of Excess Remittances | 3 478.92 | | |
| Investment Fund | 18 349.70 | | |
| Book of Standards Reserve | 119 370.16 | | |
| Technical Assistant Committee C-1 | 1 824.00 | | |
| Transfer to Joint Committee on X-ray Diffraction | 59 500.00 | | |
| Cost and Refunds on X-ray Diffraction Cards | 34 763.67 | | |
| Institute of Petroleum Publications | 11.06 | | |
| Annual Meeting Luncheon | 1 529.20 | | |
| Annual Meeting Buffet Supper | 353.50 | | |
| Executive Retirement Reserve | 900.00 | | |
| Employee Retirement Reserve | 198.86 | | |
| Gillett Memorial Fund | 415.45 | | |
| Notes Receivable Investment Fund | 55 000.00 | | |
| Special Annual Meeting Account | 1 000.00 | | |
| Pacific Coast Meeting | 1 371.79 | | |
| Charge for Bad Debts | 25.85 | | |
| Miscellaneous | 213.35 | | |
| Total Disbursements | | 298 305.51 | |
| Total Disbursements | | | 1 194 819.00 |
| Cash Balance, November 30, 1956 | | | $126 447.80 |

[b] ( ) Indicate red figures

## ASTM BUILDING FUND

### Details of ASTM Building Fund Cash Receipts and Disbursements

### For the Period December 11, 1955 to November 3, 1956

| | | | |
|---|---|---|---|
| Cash balance, December 10, 1955 | | | $8 942.28 |
| *Receipts* | | | |
| Contributions from Members | | $3 135.52 | |
| Interest and Dividends and Profit on Investments | | 1 623.83 | |
| From ASTM General Fund: | | | |
| On Account of Depreciation on Building, Elevator, and Air Conditioning Installation | | 9 700.00 | |
| On Account of Amortization of Note | | 7 975.00 | 22 434.35 |
| | | | 31 376.63 |
| *Disbursements* | | | |
| Expenses of Cherry St. Property: | | | |
| Real Estate Taxes (1955–1956) | 4 713.99 | | |
| Air Conditioner | 282.15 | 4 996.14 | |
| Expenses of 1916 Race St.: | | | |
| Asphalt Tile | 528.00 | | |
| Air Conditioners | 515.00 | 1 043.00 | |
| Transferred to Investment Fund | | 715.74 | |
| Reduction to Loan from ASTM General Fund | | 7 975.00 | 14 729.88 |
| Cash balance, November 30, 1956 | | | $16 646.75 |

## ASTM BUILDING FUND

### DETAILS OF ASSETS AND LIABILITIES OF BUILDING FUND AS OF NOVEMBER 30, 1956

*Assets*

| | | | | | |
|---|---|---|---|---|---|
| Cash in Girard Trust Corn Exchange Bank, Philadelphia, Pa. | | | | | $16 646.75 |
| Investments Fund Participation | | | | | 20 961.02 |
| Property 1916–1918 Race St., Philadelphia, Pa.: | | | | | |
| Cost: | | | | | |
| Land | | | | 30 000.00 | |
| Building | | $26 503.35 | | | |
| Construction (Net of Salvage) to 12/10/55 | | $134 288.06 | | | |
| Additions 1956: Floor Tile | | 528.00 | | | |
| Air Conditioner | | 515.00 | 161 834.41 | | |
| Accumulated Depreciation (Provided from General Funds) | | | 54 283.05 | 107 551.36 | |
| | | | | 137 551.36 | |
| Property 1921–1927 Cherry St., and 1922–1928 Quarry St., Philadelphia, Pa.: | | | | | |
| Cost: | | | | | |
| Land | | | 30 800.00 | | |
| Buildings | 38 880.10 | | | | |
| Construction | 62 898.28 | | | | |
| Additions 1956: | | | | | |
| Air Conditioner | 282.15 | | | | |
| | 102 060.53 | | | | |
| Accumulated Depreciation (Provided from General Funds) | | 9 516.95 | 92 543.58 | 123 343.58 | |
| Property 1919 Cherry St., Philadelphia, Pa.: | | | | | |
| Cost, land | | | 7 659.42 | | |
| Improvements | | | 890.47 | 8 549.89 | 269 444.83 |
| Total assets | | | | | 307 052.60 |

*Liabilities and Funds*

| | | | | |
|---|---|---|---|---|
| Contributions from Members | | | $181 054.42 | |
| Contributions from ASTM Funds | | | 73 364.57 | |
| Accumulated Income: | | | | |
| Interest and Dividends on Investments | | 7 940.77 | | |
| Profit on Disposal of Securities, net | | 869.96 | | |
| Profit on Sale of 1915–1918 Cherry St. | | 10 502.49 | | |
| Rental on Cherry St. Property | | 778.92 | | |
| | | 20 092.14 | | |
| Less Heating of Cherry St. Property | $812.50 | | | |
| Interest on Loan from General Fund | 4 010.96 | | | |
| Real Estate Taxes on Cherry St. | 7 599.15 | | | |
| Water and Sewerage on Cherry St. | 126.23 | | | |
| Moving Expense | 1 309.69 | | | |
| Appraisal | 325.00 | 14 183.53 | 5 908.61 | |
| Loan Payable to ASTM General Funds | | | 46 725.00 | |
| | | | | $307 052.60 |

## ASTM INVESTMENT FUND PARTICIPATIONS

### AS OF NOVEMBER 30, 1956

| | |
|---|---|
| Cash | $28 463.54 |
| Investments (at cost)<br>(Market value $871 704.54) | 708 759.75 |
| Accrued Interest (on bonds purchased 1956) | 599.36 |
| Total assets | $737 822.65 |

| Participations by funds, per cent: | | 12/10/55 *Participations* | *Income Reinvested* | *Net Profit Sale of Securities* | *Total Participation 11/30/56 (At Cost)* |
|---|---|---|---|---|---|
| General Fund | 72.83433 | $519 038.45 | $4 289.77 | $14 059.93 | $537 388.15 |
| Committee Funds | 13.57964 | 96 772.45 | 799.81 | 2 621.41 | 100 193.67 |
| Research Fund | 9.12649 | 65 037.97 | 537.53 | 1 761.78 | 67 337.28 |
| Building Fund | 2.84093 | 20 245.28 | 167.33 | 548.41 | 20 961.02 |
| Medal & Lecture Fund | .94105 | 6 706.25 | 55.42 | 181.66 | 6 943.33 |
| Templin Award Fund | .30114 | 2 146.00 | 17.74 | 58.13 | 2 221.87 |
| Sam Tour Fund | .23437 | 1 670.24 | 13.80 | 45.24 | 1 729.28 |
| Richart Fund | .14205 | 1 012.26 | 8.37 | 27.42 | 1 048.05 |
| | 100.00000 | $712 628.90 | $5 889.77 | $19 303.98 | $737 822.65 |

## ASTM INVESTMENT FUND

### DETAILS OF INVESTMENT FUND CASH RECEIPTS AND DISBURSEMENTS

### FOR THE PERIOD DECEMBER 11, 1955 TO NOVEMBER 30, 1956

| | | | |
|---|---|---|---|
| Cash Balance, December 10, 1955 | | | $165 300.83 |
| *Receipts* | | | |
| Interest and Dividends on Investment | $33 753.07 | | |
| Interest on Savings Accounts | 3 039.56 | | |
| Interest on Certificates of Deposit | 1 712.50 | | |
| Total Interest and Dividends | | $38 505.13 | |
| Proceeds from Sales of Investments | 89 651.23 | | |
| Loan from General Funds | 55 000.00 | | |
| Cash Received in Exchange of Allegheny Ludlum Steel | 14.08 | 144 665.31 | |
| Total receipts | | | 183 170.44 |
| | | | 348 471.27 |
| *Disbursements* | | | |
| Purchase of Securities | | 231 302.34 | |
| Investment Counsel | | 1 250.00 | |
| Repayment of Loan from General Funds | | 55 000.00 | |
| Distribution of Interest and Dividends to Participants: | | | |
| General Fund | 23 638.66 | | |
| Committee Funds | 4 407.33 | | |
| Research Fund | 2 962.04 | | |
| Building Fund | 922.04 | | |
| Medal and Lecture Fund | 305.42 | | |
| Templin Award Fund | 97.73 | | |
| Sam Tour Fund | 76.06 | | |
| Richart Fund | 46.11 | 32 455.39 | |
| Total disbursements | | | 320 007.73 |
| Cash balance, November 30, 1956 | | | 28 463.54 |
| Balance composed of: | | | |
| Demand account | | 4 179.65 | |
| Savings accounts | | 14 283.89 | |
| Certificates of deposit | | 10 000.00 | $28 463.54 |

## DISBURSEMENTS

| | | | |
|---|---|---|---|
| *Operating Disbursements (Budgeted):* | | | |
| *Publications:* | | | |
| Year Book | 13 075.92 | | |
| 1952 Book of Standards | 126.65 | | |
| 1955 Book of Standards | 19 021.41 | | |
| 1953 and 1954 Supplements to Book of Standards | (47.62)[b] | | |
| 1956 Supplement to Book of Standards | 50 375.72 | | |
| Methods of Chemical Analysis of Metals | 1 284.18 | | |
| Compilation of Standards | 37 741.60 | | |
| Separate Standards | 14 817.25 | | |
| Proceedings—repurchase of old numbers | 92.00 | | |
| 1955–1956 Proceedings | 41 176.75 | | |
| Preprints | 10 903.80 | | |
| ASTM BULLETIN | 45 426.51 | | |
| Circulars to members | 4 613.85 | | |
| Index to Standards | 10 475.02 | | |
| Special and Miscellaneous Publications | 93 229.30 | | |
| West Coast—Papers and Preprints | 10.76 | | |
| Total Disbursements—Publications | | $342 323.10 | |
| Salaries | 309 401.96 | | |
| General Office Expenses | 111 557.61 | | |
| Expenses—Technical and District Committees | 11 752.62 | | |
| Expenses—Meetings | 30 453.25 | | |
| Headquarters Occupancy Expense (includes $9 700 depreciation on building) | 35 581.98 | | |
| Transfer to Principal of Research Fund | 3 200.00 | | |
| American Standards Association | 1 500.00 | | |
| Traveling Expense, Administrative and Special Committees | 8 502.99 | | |
| Furniture and Fixtures | 8 127.99 | | |
| Dues, Contributions, Miscellaneous | 1 508.75 | | |
| Group Insurance | 813.15 | | |
| Employees' Retirement Fund | 23 530.49 | | |
| Federal Old Age Survivors | 4 886.73 | | |
| Blue Cross | 1 235.46 | | |
| Major Medical | 2 137.41 | 554 190.39 | |
| Total Operating Disbursements (Budgeted) | | 896 513.49 | |
| *Nonoperating Disbursements (Not Budgeted):* | | | |
| Refund of Excess Remittances | 3 478.92 | | |
| Investment Fund | 18 349.70 | | |
| Book of Standards Reserve | 119 370.16 | | |
| Technical Assistant Committee C-1 | 1 824.00 | | |
| Transfer to Joint Committee on X-ray Diffraction | 59 500.00 | | |
| Cost and Refunds on X-ray Diffraction Cards | 34 763.67 | | |
| Institute of Petroleum Publications | 11.06 | | |
| Annual Meeting Luncheon | 1 529.20 | | |
| Annual Meeting Buffet Supper | 353.50 | | |
| Executive Retirement Reserve | 900.00 | | |
| Employee Retirement Reserve | 198.86 | | |
| Gillett Memorial Fund | 415.45 | | |
| Notes Receivable Investment Fund | 55 000.00 | | |
| Special Annual Meeting Account | 1 000.00 | | |
| Pacific Coast Meeting | 1 371.79 | | |
| Charge for Bad Debts | 25.85 | | |
| Miscellaneous | 213.35 | | |
| Total Disbursements | | 298 305.51 | |
| Total Disbursements | | | 1 194 819.00 |
| Cash Balance, November 30, 1956 | | | $126 447.80 |

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[b] ( ) Indicate red figures

## ASTM BUILDING FUND

### DETAILS OF ASTM BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS
### FOR THE PERIOD DECEMBER 11, 1955 TO NOVEMBER 3, 1956

| | | | |
|---|---|---|---|
| Cash balance, December 10, 1955 | | | $8 942.28 |
| *Receipts* | | | |
| Contributions from Members | | $3 135.52 | |
| Interest and Dividends and Profit on Investments | | 1 623.83 | |
| From ASTM General Fund: | | | |
| On Account of Depreciation on Building, Elevator, and Air Conditioning Installation | | 9 700.00 | |
| On Account of Amortization of Note | | 7 975.00 | 22 434.35 |
| | | | 31 376.63 |
| *Disbursements* | | | |
| Expenses of Cherry St. Property: | | | |
| Real Estate Taxes (1955–1956) | 4 713.99 | | |
| Air Conditioner | 282.15 | 4 996.14 | |
| Expenses of 1916 Race St.: | | | |
| Asphalt Tile | 528.00 | | |
| Air Conditioners | 515.00 | 1 043.00 | |
| Transferred to Investment Fund | | 715.74 | |
| Reduction to Loan from ASTM General Fund | | 7 975.00 | 14 729.88 |
| Cash balance, November 30, 1956 | | | $16 646.75 |

## ASTM BUILDING FUND

### DETAILS OF ASSETS AND LIABILITIES OF BUILDING FUND AS OF NOVEMBER 30, 1956

*Assets*

| | | | | | |
|---|---|---|---|---|---|
| Cash in Girard Trust Corn Exchange Bank, Philadelphia, Pa. | | | | | $16 646.75 |
| Investments Fund Participation | | | | | 20 961.02 |
| Property 1916–1918 Race St., Philadelphia, Pa.: | | | | | |
| Cost: | | | | | |
| Land | | | | 30 000.00 | |
| Building | | $26 503.35 | | | |
| Construction (Net of Salvage) to 12/10/55 | | $134 288.06 | | | |
| Additions 1956: Floor Tile | | 528.00 | | | |
| Air Conditioner | | 515.00 | 161 834.41 | | |
| Accumulated Depreciation (Provided from General Funds) | | | 54 283.05 | 107 551.36 | |
| | | | | 137 551.36 | |
| Property 1921–1927 Cherry St., and 1922–1928 Quarry St., Philadelphia, Pa.: | | | | | |
| Cost: | | | | | |
| Land | | | 30 800.00 | | |
| Buildings | 38 880.10 | | | | |
| Construction | 62 898.28 | | | | |
| Additions 1956: | | | | | |
| Air Conditioner | 282.15 | | | | |
| | 102 060.53 | | | | |
| Accumulated Depreciation (Provided from General Funds) | | 9 516.95 | 92 543.58 | 123 343.58 | |
| Property 1919 Cherry St., Philadelphia, Pa.: | | | | | |
| Cost, land | | | 7 659.42 | | |
| Improvements | | | 890.47 | 8 549.89 | 269 444.83 |
| Total assets | | | | | 307 052.60 |

*Liabilities and Funds*

| | | | | |
|---|---|---|---|---|
| Contributions from Members | | | $181 054.42 | |
| Contributions from ASTM Funds | | | 73 364.57 | |
| Accumulated Income: | | | | |
| Interest and Dividends on Investments | | 7 940.77 | | |
| Profit on Disposal of Securities, net | | 869.96 | | |
| Profit on Sale of 1915–1918 Cherry St | | 10 502.49 | | |
| Rental on Cherry St. Property | | 778.92 | | |
| | | 20 092.14 | | |
| Less Heating of Cherry St. Property | $812.50 | | | |
| Interest on Loan from General Fund | 4 010.96 | | | |
| Real Estate Taxes on Cherry St. | 7 599.15 | | | |
| Water and Sewerage on Cherry St. | 126.23 | | | |
| Moving Expense | 1 309.69 | | | |
| Appraisal | 325.00 | 14 183.53 | 5 908.61 | |
| Loan Payable to ASTM General Funds | | | 46 725.00 | |
| | | | | $307 052.60 |

## ASTM INVESTMENT FUND PARTICIPATIONS

As of November 30, 1956

| | |
|---|---|
| Cash | $28 463.54 |
| Investments (at cost) | 708 759.75 |
| (Market value $871 704.54) | |
| Accrued Interest (on bonds purchased 1956) | 599.36 |
| Total assets | $737 822.65 |

| | | 12/10/55 *Participations* | *Income Reinvested* | *Net Profit Sale of Securities* | *Total Participation* 11/30/56 *(At Cost)* |
|---|---|---|---|---|---|
| Participations by funds, per cent: | | | | | |
| General Fund | 72.83433 | $519 038.45 | $4 289.77 | $14 059.93 | $537 388.15 |
| Committee Funds | 13.57964 | 96 772.45 | 799.81 | 2 621.41 | 100 193.67 |
| Research Fund | 9.12649 | 65 037.97 | 537.53 | 1 761.78 | 67 337.28 |
| Building Fund | 2.84093 | 20 245.28 | 167.33 | 548.41 | 20 961.02 |
| Medal & Lecture Fund | .94105 | 6 706.25 | 55.42 | 181.66 | 6 943.33 |
| Templin Award Fund | .30114 | 2 146.00 | 17.74 | 58.13 | 2 221.87 |
| Sam Tour Fund | .23437 | 1 670.24 | 13.80 | 45.24 | 1 729.28 |
| Richart Fund | .14205 | 1 012.26 | 8.37 | 27.42 | 1 048.05 |
| | 100.00000 | $712 628.90 | $5 889.77 | $19 303.98 | $737 822.65 |

## ASTM INVESTMENT FUND

### Details of Investment Fund Cash Receipts and Disbursements

### For the Period December 11, 1955 to November 30, 1956

| | | | |
|---|---|---|---|
| Cash Balance, December 10, 1955 | | | $165 300.83 |

*Receipts*

| | | | |
|---|---|---|---|
| Interest and Dividends on Investment | $33 753.07 | | |
| Interest on Savings Accounts | 3 039.56 | | |
| Interest on Certificates of Deposit | 1 712.50 | | |
| Total Interest and Dividends | | $38 505.13 | |
| Proceeds from Sales of Investments | 89 651.23 | | |
| Loan from General Funds | 55 000.00 | | |
| Cash Received in Exchange of Allegheny Ludlum Steel | 14.08 | 144 665.31 | |
| Total receipts | | | 183 170.44 |
| | | | 348 471.27 |

*Disbursements*

| | | | |
|---|---|---|---|
| Purchase of Securities | | 231 302.34 | |
| Investment Counsel | | 1 250.00 | |
| Repayment of Loan from General Funds | | 55 000.00 | |
| Distribution of Interest and Dividends to Participants: | | | |
| General Fund | 23 638.66 | | |
| Committee Funds | 4 407.33 | | |
| Research Fund | 2 962.04 | | |
| Building Fund | 922.04 | | |
| Medal and Lecture Fund | 305.42 | | |
| Templin Award Fund | 97.73 | | |
| Sam Tour Fund | 76.06 | | |
| Richart Fund | 46.11 | 32 455.39 | |
| Total disbursements | | | 320 007.73 |
| Cash balance, November 30, 1956 | | | 28 463.54 |
| Balance composed of: | | | |
| Demand account | | 4 179.65 | |
| Savings accounts | | 14 283.89 | |
| Certificates of deposit | | 10 000.00 | $28 463.54 |

## INVESTMENT FUND

### AS OF NOVEMBER 30, 1956

### *Investments*

NOTE: More than the usual number of transactions took place during the year. However new investment counsel recommended some shifting to place the holdings of the Society on a basis more readily compatible with what seemed prudent considering the economic outlook.

| *Number of Shares or Par Value* | | *Cost or Book Value* | *Market Value*[a] |
|---|---|---|---|
| | *Bonds* | | |
| 5 000 | American Tobacco Debs. 3s 10/15/69 | 5 067.08 | 4 500.00 |
| 15 000 | Consolidated Edison Co., N. Y., Inc. 1st & Ref. 3⅝ per cent Ser. L5/1/86 | 15 206.55 | 14 100.00 |
| 10 000 | Detroit Edison Co. 3¾ per cent Con. Deb. 9/14/71 | 11 000.00 | 10 900.00 |
| 15 000 | General Motors Corp. 3¼ Deb. 1/1/79 | 14 925.00 | 14 006.25 |
| 1 000 | General Telephone Corp. 4 per cent Con. Deb. 5/1/71 | 1 001.25 | 1 005.00 |
| 5 000 | Pacific Gas & Electric Co. 1st & Ref. 3⅛s Ser. "W" 12/1/84 | 5 150.78 | 4 800.00 |
| 10 000 | Radio Corp. of America 3½ per cent Con. Deb. 12/1/80 | 11 075.00 | 9 500.00 |
| 10 000 | Scott Paper Co. 3 per cent Con. Deb. 3/1/71 | 10 977.80 | 9 500.00 |
| 15 000 | Socony Mobil Co. 2½s Debs. 6/1/76 | 13 995.95 | 12 750.00 |
| 15 000 | Southern Natural Gas Co. 1st Mtg. Pipe Line S F 3⅛s 2/1/74 | 14 734.81 | 13 500.00 |
| 15 000 | Superior Oil Co. 3¾ per cent Deb. 7/1/81 | 15 000.00 | 14 475.00 |
| 10 000 | Tennessee Gas Transmission Co. 1st Mtg Pipeline 4¾s 11/1/76 | 10 065.00 | 9 925.00 |
| 5 000 | U. S. Federal Land Banks 3⅞ per cent 9/15/72 | 4 975.00 | 4 981.25 |
| 5 000 | U. S. Twelve Fed. Land Banks 3¾ per cent 7/15/57 | 5 000.00 | 5 001.56 |
| 15 000 | U. S. Twelve Fed. Land Banks 2¾ per cent 5/1/58 | 14 850.00 | 14 709.38 |
| 2 000 | U. S. Treasury 2½s 6/15/64–69 | 2 000.00 | 1 800.00 |
| 5 000 | U. S. Treasury 2½s 3/15/65–70 | 5 000.00 | 4 493.75 |
| 15 000 | U. S. Treasury 2½s 11/15/61 | 15 000.00 | 14 217.19 |
| 2 000 | U. S. Treasury 3¼s 6/15/78–83 | 2 000.00 | 1 966.25 |
| 3 000 | U. S. Treasury 2½s 12/15/67–72 | 3 000.00 | 2 681.25 |
| 8 500 | U. S. Treasury 2½s 6/15/67–72 | 8 632.83 | 7 602.19 |
| 15 000 | U. S. Treasury 2½s 8/15/63 | 14 943.75 | 14 062.50 |
| 15 000 | U. S. Treasury 2¾s 9/15/61 | 14 934.38 | 14 432.81 |
| 30 000 | U. S. Savings Bonds Ser. "G" 2½s 6/1/61 | 30 000.00 | 28 920.00 |
| 5 000 | U. S. Savings Bonds Ser. "G" 2½s 1/1/62 | 5 000.00 | 4 775.00 |
| 5 000 | U. S. Savings Bonds Ser. "G" 2½s 2/1/62 | 5 000.00 | 4 775.00 |
| | Total Bonds | 258 535.18 | 243 379.38 |
| *Shares* | | | |
| | *Preferred Stocks* | | |
| 30 | Armstrong Cork Co., 3.75 per cent Cum. Pfd. Ser. "A" | 2 947.09 | 2 580.00 |
| 30 | Consolidated Edison Co. $5 Cum. Pfd. | 3 286.08 | 3 120.00 |
| 30 | Endicott Johnson Corp. 4 per cent Pfd. | 2 937.50 | 2 520.00 |
| 50 | Food Machinery & Chemical Corp. 3¼ per cent Cum. Conv. Pfd. | 4 750.10 | 6 250.00 |
| 30 | General Motors Corp. $5 Pfd. | 3 755.80 | 3 345.00 |
| 60 | Jones & Laughlin Steel Corp. $5 Cum. Pfd. Ser. "A" | 5 386.73 | 5 790.00 |
| 30 | Long Island Lighting Co. 4.35 per cent Ser. "E" Pfd. | 3 015.00 | 2 610.00 |
| 30 | P. Lorillard Co. 7 per cent Pfd | 4 380.00 | 3 750.00 |
| 30 | Mississippi Power & Light Co. | 3 075.00 | 2 550.00 |
| 100 | Philadelphia Electric Co. 4.40 per cent Cum. Pfd. | 10 670.00 | 10 050.00 |
| 47 | Standard Slag Co. 2nd Pfd. | 2 115.00 | 2 115.00 |
| 30 | Westinghouse Electric Corp. 3.80 per cent Cum. Pfd. Ser. "B" | 3 067.74 | 2 490.00 |
| | Total Preferred Stocks | 49 386.04 | 47 170.00 |

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[a] All market values of securities are as of November 26, 1956.

| Shares | | Cost or Book Value | Market Value[a] |
|---|---|---|---|
| | *Common Stocks* | | |
| 124 | Allegheny Ludlum Steel Corp. | $3 023.26 | $6 820.00 |
| 100 | Aluminum Company of America | 2 061.45 | 9 250.00 |
| 100 | American Can Co. | 3 884.25 | 4 037.50 |
| 150 | American Cyanamid Co. | 6 238.91 | 10 743.75 |
| 100 | American Radiator & Standard Sanitary Co. | 1 564.32 | 1 762.50 |
| 158 | American Stores Co. | 7 634.13 | 8 058.00 |
| 56 | American Telephone & Telegraph Co. | 8 919.72 | 9 408.00 |
| 100 | American Tobacco Co. | 7 370 97 | 7 212.50 |
| 150 | Atchison, Topeka & Sante Fe R.R. Co. | 2 961.06 | 3 937.50 |
| 40 | Bethlehem Steel Corp. | 2 095.99 | 6 830.00 |
| 230 | Carpenter Steel Co. | 6 104.75 | 12 707.50 |
| 136 | Chase Manhattan Bank | 3 929.50 | 6 562.00 |
| 200 | Cleveland Electric Illuminating Co. | 8 271.00 | 8 050.00 |
| 150 | Climax Molybdenum Co. | 8 879.80 | 9 750.00 |
| 100 | Crown Zellerbach Corp. | 2 914.79 | 5 087.50 |
| 75 | Delaware Light & Power Co. | 2 454.11 | 3 187.50 |
| 167 | Dow Chemical Co. | 3 997.75 | 11 335.13 |
| 30 | E. I. du Pont de Nemours & Co. | 2 899.12 | 5 422.50 |
| 50 | Florida Power & Light Co. | 1 969.44 | 2 231.25 |
| 125 | General Electric Co. | 4 311.15 | 7 343.75 |
| 150 | General Mills, Inc. | 10 630.89 | 9 675.00 |
| 225 | General Telephone Corp. | 3 312.82 | 8 746.88 |
| 50 | B. F. Goodrich Co. | 1 647.31 | 3 568.75 |
| 100 | Great Northern Paper Co. | 10 045.00 | 8 025.00 |
| 129 | Gulf Oil Corp. | 3 476.68 | 13 351.50 |
| 206 | Houston Lighting & Power Co. | 7 112.18 | 10 789.25 |
| 200 | International Harvester Co. | 6 406.67 | 7 400.00 |
| 110 | Johns-Manville Corp. | 3 833.25 | 5 225.00 |
| 100 | Jones & Laughlin Steel Corp. | 2 636.06 | 5 825.00 |
| 200 | Lone Star Cement Corp. | 4 941.44 | 17 000.00 |
| 50 | Louisville & Nashville R.R. Co. | 4 154.59 | 4 437.50 |
| 200 | Montgomery Ward & Co. | 5 798.04 | 8 050.00 |
| 103 | Niagara Mohawk Power Corp. | 3 479.57 | 3 077.13 |
| 200 | Ohio Oil Co. | 2 623.35 | 8 550.00 |
| 60 | Pacific Gas & Electric Co. | 2 301.42 | 2 940.00 |
| 50 | Parke, Davis & Co. | 1 641.19 | 2 175.00 |
| 321 | Philadelphia Electric Co. | 8 599.20 | 11 716.50 |
| 100 | Republic Steel Corp. | 2 152.93 | 5 700.00 |
| 200 | Seaboard Oil Co | 11 956.88 | 13 900.00 |
| 152 | Sears, Roebuck & Co. | 3 155.22 | 4 503.00 |
| 100 | Sherwin Williams Co. | 10 515.69 | 11 500.00 |
| 125 | Socony Mobil Oil Co. | 2 510.28 | 6 671.88 |
| 150 | Square D Co. | 2 362.37 | 4 312.50 |
| 200 | Standard Oil of Indiana | 11 969.64 | 12 000.00 |
| 270 | Standard Oil Co. of N. J. | 8 526.48 | 14 883.75 |
| 80 | The Texas Co. | 2 184.35 | 4 390.00 |
| 60 | Union Carbide & Carbon Corp. | 4 235.24 | 6 375.00 |
| 240 | Westinghouse Electric Corp. | 15 644.26 | 12 450.00 |
| 200 | Wisconsin Electric Power Co. | 6 788.63 | 6 650.00 |
| | Total Common Stocks | 256 127.10 | 373 625.52 |
| *Shares* | *Mutual Funds* | | |
| 849 | Broad Street Investing Corp. | 11 337.87 | 19 161.93 |
| 4 092.505 | Commonwealth Investment Co. | 29 476.08 | 37 814.75 |
| 5 376 | Eaton Howard Balanced Fund | 83 433.04 | 116 605.44 |
| 2 176 | Fundamental Investors, Inc. | 20 464.44 | 33 947.52 |
| | Total Mutual Funds | 144 711.43 | 207 529.64 |
| | *Summary* | | |
| | Bonds | 258 535.18 | 243 379.38 |
| | Preferred Stocks | 49 386.04 | 47 170.00 |
| | Common Stocks | 256 127.10 | 373 625.52 |
| | Mutual Funds | 144 711.43 | 207 529.64 |
| | | $708 759.75 | $871 704.54 |

## APPENDIX II

## REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

The Administrative Committee on Standards is interested in all aspects of the Society's standardization work. The several functions are discussed under the subdivision headings of this report.

*Actions by Standards Committee on Committee Recommendations:*

The review of recommendations from technical committees is, of course, one of the primary functions of the Standards Committee since in its establishment it was intended to provide a means of having recommendations acted upon in the interval between Annual Meetings. Many of the annual reports refer to recommendations that have been submitted to the Standards Committee during the year. The actions taken this year by the Standards Committee are summarized below:

| | |
|---|---|
| New Tentatives | 23 |
| Revised Tentatives | 22 |
| Tentative Revisions of Standards | 7 |
| Standards Revised and Reverted to Tentative | 6 |
| Emergency Alternates Withdrawn | 4 |

A large number of these recommendations were acted upon at a meeting held on September 5, 1956; the remainder were handled by correspondence.

*Actions by the Society at 1956 Annual Meeting:*

As a matter of record, the actions taken by the Society at the 1956 Annual Meeting with respect to standards and tentatives are tabulated below:

| | |
|---|---|
| New Standard | 1 |
| Existing Tentatives Adopted as Standard | 123 |
| Standards Revised | 75 |
| New Tentatives | 83 |
| Standards Revised and Reverted to Tentative | 13 |
| Tentative Revisions of Standards | 14 |
| Tentatives Revised | 121 |
| Standards and Tentatives Withdrawn | 10 |

*Form of Standards:*

Improvement of the quality and form of standards continues to be an important consideration of the committee. Editorial committees have been organized by a number of the technical committees, and the Standards Committee would wish to see this practice extended. The recommendations covering the form of ASTM specifications referred to in previous reports is now available in printed form for the use of the technical committees. A comparable manual covering recommendations on the form of methods of chemical analysis has recently been made available, and Committee E-1 on Methods of Testing has drafted a companion manual on methods of testing in general.

### Expansion of Standardization Activities

The Standards Committee continues to explore with the Board of Directors proposals dealing with new projects

Comments with respect to a number of these are set forth below.

*Sorbent Mineral Materials.*—The Board of Directors acted favorably on the recommendation that a new Committee on Sorbent Mineral Materials be established. The organization meeting of this committee has now been held.

*Manganese Dioxide.*—The recommendation that a separate committee on manganese dioxide be established was favorably acted upon by the Board of Directors, but the organization of this committee is being held in abeyance pending further advice from the industry as to whether the present time is opportune for the organization of such a committee.

*Casein and Similar Protein Materials.*—The report of the Standards Committee a year ago referred to the authorization of a new technical committee on casein. In the organization of this committee it developed that attention should not be confined to casein alone but other similar protein materials should be included. This extension relates specifically to soybean proteins.

*Industrial Chemicals.*—As indicated in the report a year ago, the subject of industrial chemicals is so broad that considerable study is required to delineate the field more specifically. Preliminary conferences have been held and it is being recommended that one or more product committees be established in the chemicals field. A study committee is being continued and with the advice of this committee suitable conferences will be set up in specific fields.

*Cleaning Solvents.*—Requests have been received that work be undertaken in the field of cleaning solvents. Two specific applications have been mentioned: one, cleaners used for electric motors; the other, vapor degreasers used for the cleaning of metal surfaces. A conference will be called shortly and this may indicate more definitely the needs on the part of industry in these and related fields.

*Flexible Packaging Materials.*—A need has been expressed for standards, particularly test procedures, relating to flexible packaging materials. This proposal is of interest to a number of the materials committees and to Committee D-10 on Shipping Containers. The subject, however, would appear to be too broad for any of the present committees. A conference is being called which would include a number of representatives from the industry in addition to representatives from the various ASTM committees concerned to resolve whether a new technical committee is needed. The function of such a committee might be largely of a coordinating nature.

*Coal Tar Resins.*—In view of the need expressed for methods of testing color of coal tar resins, new work has been undertaken under Committee E-12 on Appearance. It is recognized that there may be needed test procedures other than for color and other appearance factors, and this work may ultimately be broadened to such an extent that a separate committee might be indicated.

*Cermets.*—The so-called cermets have come up for consideration in several committees of the Society including the Joint Committee on Effect of Temperature on the Properties of Metals. A conference of representatives of these several committees held during the year recommended that a study committee be appointed to determine whether any standardization in this field is desirable. One of the problems before this group would be to resolve with more clarity than has been done at the present time as to the particular materials that are involved under the general term of "cermets." This study committee is now in process of formation.

## RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Two methods under the procedure of the American Standards Association are used by ASTM in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship insofar as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, usually with the ASTM as sponsor or cosponsor of the sectional committee.

*Standards Submitted Under the Existing Standards Procedure:*

No standards were submitted to the American Standards Association for approval as American Standard under the existing standards procedure.

*Revised Standards Submitted Under the Proprietary Procedure:*

In view of the Society having been designated as proprietary sponsor for the revisions of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Association during the year revisions of 5 standards relating to steel, 1 relating to cast iron, 3 relating to refractories, 1 relating to manufactured masonry units, 4 relating to bituminous roofing materials, 1 relating to pigments, 1 relating to gypsum, 4 relating to soaps, 1 relating to thermometers, and 1 relating to fire tests of door assemblies.

*Standards Submitted Under the Sectional Committee Procedure:*

During the year, the following three sectional committees, for which the Society is sponsor or cosponsor, took action to present recommendations to the ASA as follows:

*Sectional Committee A37 on Road and Paving Materials.*—This committee has recommended the approval of revisions of 21 American Standard and the reaffirmation of one American Standard.

*Sectional Committee L14 on Textile Test Methods.*—This committee has recommended the approval as American Standard of 14 ASTM standards and the approval of revisions of one American Standard.

*Sectional Committee Z11 on Petroleum Products and Lubricants.*—This committee has recommended the approval as American Standard of one ASTM standard, and the approval of revisions of 13 American Standards.

## INTERNATIONAL RELATIONS

The international discussions, particularly under the International Organization for Standardization (ISO) are assuming ever greater significance. A number of the European countries apparently wish to have the standardization discussions handled internationally and much of the work is supported here rather than in the development of individual national standards. In certain areas these international discussions are having a definite impact on the standardization in this country and a number of ASTM committees are following these international discussions quite closely with the thought that the ASTM standards and the international recommendations should be in harmony. A list of the various ISO projects in which ASTM is interested and which continues to grow year by year is given below.

*Aromatic Hydrocarbons.*—ASTM Committee D-16 is giving consideration to American participation in the work of ISO/TC 78 on Aromatic Hydrocarbons for which Great Britain holds the secretariat.

*Asbestos-Cement Products.*—ASTM Committee C-17 on Asbestos-Cement Products has indicated that it favors American participation in the work of ISO/TC 77 on the basis of an observer. Switzerland holds the secretariat for this ISO committee.

*Cast Iron.*—Great Britain holds the secretariat for ISO/TC 25 on Cast Iron. The Advisory Committee of ASTM Committee A-3 has been set up to handle American participation.

*Chemistry.*—Italy holds the secretariat for ISO/TC 47 on Chemistry. Several ASTM committees have indicated an interest in this work, and draft proposals which have been received have been passed on to these committees.

*Copper and Copper Alloys.*—The United States holds the secretariat for ISO/TC 26 on Copper and Copper Alloys. The Advisory Committee of ASTM Committee B-5 has been set up to handle the work of the secretariat.

*Iron and Steel.*—The United States has the status of an observer in the work of ISO/TC 17 on Iron and Steel for which Great Britain holds the secretariat. A number of draft proposals have been received which have been brought to the attention of Committee A-1 on Steel, while others dealing with methods of testing have been submitted to the appropriate subcommittees of Committee E-1 on Methods of Testing.

*Laboratory Glassware.*—Great Britain holds the secretariat for ISO/TC 48 on Laboratory Glassware. America is participating in this work through a special group on which ASTM is represented through Committee E-1 on Methods of Testing.

*Light Metals and Alloys.*—The Advisory Committee of ASTM Committee B-7 has been set up to handle American participation in the work of ISO/TC 79 on Light Metals and Alloys. France holds the secretariat for this ISO committee.

*Mica.*—Subcommittee IX on Mica of ASTM Committee D-9 has been set up to handle the American contacts with ISO/TC 56 on Mica for which India holds the secretariat.

*Paper.*—On the recommendation of ASTM Committee D-6, America participates in the work of ISO/TC 6 on Paper on the basis of an observer. France holds the secretariat for this ISO committee.

*Petroleum Products.*—The United States holds the secretariat for ISO/TC 28 on Petroleum Products, the technical work of the secretariat being under the purview of Sectional Committee Z11 on Petroleum Products and Lubricants, for which ASTM is sponsor.

*Plastics.*—America holds the secretariat for ISO/TC 61 on Plastics. ASTM Committee D-20 on Plastics is handling the work of the secretariat and has organized for the purpose a special committee consisting of representatives of the various groups interested.

*Raw Materials for Paints, Varnishes, and Similar Products.*—ASTM Committee D-1 is handling American contacts with ISO/TC 35 on Raw Materials for Paints, Varnishes, and Similar Products, for which the Netherlands holds the secretariat.

*Refractories.*—ASTM Committee C-8 is handling American contacts with ISO/TC 33 on Refractories for which Great Britain holds the secretariat.

*Rubber.*—The secretariat for ISO/TC 45 on Rubber is held by Great Britain. The American group to handle participation in this ISO committee has been set up under ASTM Committee D-11.

*Shellac.*—Subcommittee XIII on Shellac of ASTM Committee D-1 has been set up as the American committee in charge of contacts with ISO/TC 50 on Lac for which India holds the secretariat.

*Solid Mineral Fuels.*—ASTM Committee D-5 is handling the contacts of the United States with ISO/TC 27 on Solid Mineral Fuels for which Great Britain holds the secretariat.

*Statistical Treatment of Series of Observations.*—The Netherlands holds the secretariat for ISO/TC 69 on Statistical Treatment of Series of Observations. America is participating in this work through a special group on which ASTM is represented.

*Textiles.*—Great Britain holds the general secretariat for ISO/TC 38 on Textiles but the United States holds the secretariat for several subcommittees. American participation in this work is being handled by a special committee organized under the sponsorship of ASTM and the American Association of Textile Chemists and Colorists with representation from the various groups interested. This special committee has been given the designation of L23.

*Timber.*—ASTM Committee D-7 on Wood has indicated that it favors American participation in the work of ISO/TC 55 on Timber for which Russia holds the secretariat.

*Viscosity.*—The United States holds the secretariat for ISO/TC 66 on Viscosity. The Subcommittee on Rheological Properties of ASTM Committee E-1 has been set up to handle the work of the secretariat.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
*Chairman.*

R. E. HESS,
*Secretary.*

## APPENDIX III

## REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

A meeting of this committee was held October 31, 1956—at ASTM Headquarters.

W. J. Smith was appointed for a three-year term in June to replace H. H. Lester, deceased. W. H. Lutz was also appointed for a three-year term in June, 1956.

A successful Symposium on Titanium was promoted by the Administrative Committee in cooperation with Committee B-2 on Non-Ferrous Metals and Alloys. This was given at the Los Angeles meeting in September, 1956. A symposium on research is being planned for the 1958 annual meeting in Boston.

The publication of a series of short articles—"ACR Notes"—in the ASTM BULLETIN was continued throughout the year. Each member of the committee contributed at least one article. Attention continues to be given to securing articles of a popular nature decribing researches of probable interest to BULLETIN readers. Since one of the functions of the ACR is to help disseminate information, considerable attention has been given to devising means whereby the BULLETIN may be made more useful and interesting.

An activity assumed by the ACR some years ago was that of calling attention to technical problems where work or information is desired by the various committees of the Society. These "Unsolved Problems" have been brought to the attention of educational institutions and others to stimulate research in the fields suggested. This activity has undergone considerable study in the past two years, one result of which has been to change the designation to the more definitive term, "Challenges in Materials Research." Another has been to outline a plan for encouraging students in engineering materials to work on some of the problems that have been suggested. This conceivably could be coordinated with ASTM District activities in encouraging and rewarding outstanding engineering students who take an interest in ASTM affairs.

An effort is being made to have each of the technical committees appoint a small subcommittee or designate one or more individuals to follow for the committee its research affairs. These would be useful in finding, calling attention to, and following any research or challenges in materials research of particular interest to the committee. They would also aid in compiling information on research activity for the ASTM Staff, as well as for the ACR. Closely associated with this request has been the request of May 3, 1956, to officers of technical committees to incorporate a specific section on research in their annual reports. Such a section, incidentally, could be readily compiled into material for an ASTM "Review of Research."

Active liaison with the National Research Council was maintained during the year. Also, the work of various committees involving research has been reviewed by the ACR. These include work of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals, the Joint Committee on Chemical Analysis by Powder Diffraction Methods, Committee C-16 on Thermal Insulating Materials, Committee D-7 work on Wood Pole Research, Committee E-5 work on Fire Tests of Materials and Construction, and work of the Advisory Committee on Corrosion.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
*Chairman.*

FRANK Y. SPEIGHT,
*Secretary.*

## APPENDIX IV

## REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The year 1956 has been an extremely heavy one for the Committee on Papers and Publications. The one item that stands out is the West Coast meeting since there were upwards of 200 papers presented at that meeting in some 43 sessions. While not all of these papers were intended for publication, nevertheless these were the exception and a detailed review was required of all of those papers where publication was a possibility. For the most part the papers are being published in separate technical publications and these are listed below under a separate heading "Second Pacific Area Meeting Papers" under "Special Technical Publications in Prospect." Some few of the papers have appeared in the ASTM BULLETIN.

Apart from the West Coast papers, however, the 1956 Annual Meeting contributed its usual quota of publications including a *Proceedings* somewhat larger than the several preceding years; and there have been several other special meetings and symposiums which will result in sizable publications. Of special interest in this connection is a meeting sponsored jointly with several other organizations for the presentation of papers recently released by the AEC covering Nondestructive Testing in the Nuclear Field. A rather extensive symposium was sponsored by Committee D-2 on Composition of Petroleum Oils.

*1957 Annual Meeting:*

The program in prospect for the 1957 Annual Meeting includes the following symposiums:

Symposium on Determination of Gases in Metals,
Symposium on Spectrochemical Analysis for Trace Elements,
Symposium on Large Fatigue Testing Machines and Their Results,
Symposium on Radiation Effects on Materials, and
Symposium on Determination of Dissolved Oxygen in Water

*Book of ASTM Standards:*

In printing the 1955 Book of ASTM Standards in seven parts, two of the parts were oversize in the sense that they required hand binding rather than machine binding. This was so despite the fact that special thin paper was used in printing the Book of Standards. While it is physically feasible to have larger books, it is not considered desirable from the point of view of handling. On the Board's instructions the committee is accordingly looking into a different division of the Book of Standards so as to make each of the parts of reasonable size and such that none will be beyond the maximum size for the next two or three editions. The advice of technical committees has been sought as to the amount of material that can be expected during the next six to nine years. Also suggestions have been solicited concerning the best subdivision from a utility point of view. With this information as background, recommendations are now being formulated for the consideration of the Board. Ten parts will probably be needed in place of the present seven.

*Year Book:*

As mentioned in the report a year ago, the publication of the Year Book creates quite a problem in view of the ever increasing size of the list of members, but more particularly the personnel of the technical committees and subcommittees. Several sections were eliminated that had previously been published such as Regulations Governing Technical Committees and the various administrative committees and the geographic distribution of members. Each of these omissions has created some hardship, and consideration is being given to the reinsertion of this material. This, of course, will make the publication of the Year Book even a greater problem. Studies are being continued to see if the material in the Year Book can be published on some different basis.

## Record of Publications Issued During the Year

*Regular Publications:*

1956 Proceedings, 1528 pp., 7000 copies.
1956 Marburg Lecture, 51 pp., 2500 copies.
1956 Gillett Lecture, 32 pp., 2500 copies.
1956 Supplements to Book of Standards:
  Part 1, 474 pp., 17,000 copies.
  Part 2, 351 pp., 13,000 copies.
  Part 3, 349 pp., 11,000 copies.
  Part 4, 238 pp., 8,800 copies.
  Part 5, 334 pp., 9,000 copies.
  Part 6, 374 pp., 9,200 copies.
  Part 7, 215 pp., 8,800 copies.
Methods for Chemical Analysis of Metals, 640 pp., 7500 copies.
1956 Year Book, 640 pp., 6250 copies.
1956 Index to Standards, 240 pp., 26,500 copies.
ASTM Bulletin, 8 issues (May 1956 to April 1956), total number of pages 836, average number of copies 13,562 (232 pages of technical papers).

A statement of the volume of the principal publications for the past four years is given below:

| | 1953 to 1954 Pages | 1954 to 1955 Pages | 1955 to 1956 Pages | 1956 to 1957 Pages |
|---|---|---|---|---|
| Proceedings | 1209 | 1425 | 1128 | 1528 |
| Marburg Lecture* | .... | .... | 54 | 51 |
| Gillett Lecture* | .... | .... | .... | 32 |
| Book of ASTM Standards | .... | .... | 11752 | .... |
| Supplements to Book of Standards | 2099 | 2746 | .... | 2335 |
| ASTM Methods of Chemical Analysis of Metals | .... | .... | .... | 640 |
| Index to ASTM Standards | 300 | 306 | 216 | 240 |
| Year Book | 644 | 672 | 620 | 640 |
| Special Technical Publications | 2604 | 2101 | 3018 | 3441 |
| Other Special Publications | 938 | 46 | 964 | 878 |
| ASTM Bulletin | 724 | 792 | 808 | 836 |
| Totals | 8518 | 8088 | 18560 | 10538 |

* Issued only as a separate pamphlet from 1956 on.

*Special Compilations of Standards Published from April 15, 1956 to April 15, 1957:*

ASTM Standards on Corrosion of Iron and Steel (A-5), 144 pp., 1000 copies.
ASTM Standards on Cement (C-1), 272 pp., 2500 copies.
ASTM Standards on Petroleum Products and Lubricants (D-2), 1096 pp., 8500 copies.
ASTM Standards on Electrical Insulating Materials (D-9), 656 pp., 1000 copies. Available May, 1956—New edition in prospect.
ASTM Standards on Rubber and Rubber-Like Materials (D-11), 760 pp., 2400 copies. Available July, 1956—New edition in prospect.
ASTM Standards on Textile Materials (D-13), 836 pp., 2200 copies.
ASTM Standards on Engine Antifreezes (D-15), 64 pp., 1200 copies.
ASTM Standards on Benzene, Toluene, Xylene and Solvent Naphtha (D-16), 64 pp. 2000 copies issued in September, 1956. A second printing was struck off in January 1957—72 pp., 1000 copies.
Manual on Industrial Water (STP 148-B) (D-19), 502 pp., 1000 copies.
ASTM Standards of Plastics (D-20), 872 pp., 2500 copies.

1956 Supplement to ASTM Standards in Building Codes, 224 pp., 2000 copies.

*Special Compilations in Prospect:*

Specifications for Steel Piping Materials (A-1), 440 pp., 3000 copies.

ASTM Standards on Thermal Insulating Materials (C-16), 208 pp., 1800 copies.

ASTM Standards on Bituminous Materials for Highway Constructions, Waterproofing and Roofing (D-4, D-8), 458 pp., 2000 copies.

ASTM Standards on Rubber and Rubber-Like Materials (D-11), 810 pp., 2400 copies.

*Special Publications Issued from April 15, 1956 to April 15, 1957:*

Significance of Tests of Petroleum Products and Lubricants (STP 7-B), 133 pages, 6000 copies.

Index to the X-Ray Powder Data File (STP 48-F), 667 pp., 1000 copies.

Seventh Set of X-Ray Powder Data Cards, 600 3 x 5 white cards; 400 Keysort cards.

Bibliography and Abstracts on Electrical Contacts (STP 56-J), 45 pp., 1500 copies.

Report on Standard Samples and Related Materials for Spectrochemical Analysis (STP 58-C), 96 pp., 3000 copies.

Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (STP 150-A), 43 pp., 2000 copies.

Symposium on Evaluation of Insulating Oils—European Developments (STP 172), 78 pp., 2000 copies.

Symposium on Atmospheric Corrosion of Non-Ferrous Metals (STP 175), 164 pp., 3000 copies.

Symposium on Impact Testing (STP 176), 176 pp., 3000 copies.

Symposium on High Purity Water Corrosion (STP 179), 62 pp., 3000 copies.

Survey of Coppers—Copper Base Alloys (STP 181), 248 pp., 2500 copies.

D-11 Glossary of Terms (STP 184), 125 pp., 2000 copies.

Symposium on Speed of Testing of Non-Metallic Materials (STP 185), 92 pp., 2000 copies.

Methods for Reducing the Effect of Barometric Pressure in Measurement of Octane Number by Bruno R. Siegel (STP 186), 32 pp., 2000 copies.

Report on Relaxation Properties of Steels and Super-Strength Alloys at Elevated Temperatures (STP 187), 103 pp., 2500 copies.

Symposium on Minimum Property Values of Electrical Insulating Materials (STP 188), 54 pp., 2000 copies.

Symposium on Solder (STP 189), 196 pp., 3500 copies.

Symposium on pH Measurement (STP 190), 107 pp., 4000 copies.

Symposium on Specific Gravity of Bituminous Coated Aggregates (STP 191), 77 pp., 2000 copies.

Symposium on Steam Quality (STP 192), 55 pp., 1500 copies.

Symposium on Tension Testing of Non-Metallic Materials (STP 194), 87 pp., 2000 copies.

Symposium on Properties, Tests and Performance of Electrodeposited Metallic Coatings (STP 197), 136 pp., 3000 copies.

Symposium on Corona (STP 198), 36 pp., 2000 copies.

Elevated Temperature Properties of Wrought Medium Carbon Alloy Steels (STP 199), 127 pp., 2500 copies.

ASTM Manual for Rating Motor Fuels by Motor and Research Methods, 176 pp., 2500 copies.

Selected ASTM Engineering Materials Standards for Use in College Curricula, 370 pp., 15,000 copies.

Third Supplement to Numerical Index of Abstracted Infrared Spectra Indexed on Wyandotte ASTM Punched Cards, 12 pp., 500 copies.

Reference Photographs for Magnetic Particle Testing, 42 photographs, 3000 copies.

Summary of Proceedings, 32 pp., 8500 copies.

ASTM Five Year Index—1951–1955, 96 pp., 5000 copies.

*Special Publications in Prospect:*

1957 Supplement to the Metal Cleaning Bibliographical Abstracts (STP 90-D) 48 pp., 2000 copies.

Symposium on In-Place Shear Testing of Foundation Soil by the Vane Method (STP 193), 50 pp., 2000 copies.

Symposium on Ion-Exchange and Chromatography in Analytical Chemistry (STP 195), 90 pp., 2500 copies.

Symposium on Insulating Oil (STP 218), 45 pp., 1500 copies.

Second Pacific Area Meeting Papers:

- Papers on Metals (STP 196), 140 pp., 2500 copies.
- Symposium on Wood for Marine Use and Its Protection from Marine Organisms (STP 200), 56 pp., 2500 copies.
- Symposium on Structural Sandwich Construction (STP 201), 106 pp., 2500 copies.
- Symposium on Vapor Phase Oxidation of Gasoline (STP 202), 72 pp., 2000 copies.
- Symposium on Titanium (STP 204), 214 pp., 3000 copies.
- Papers on Cement and Concrete (STP 205), 90 pp., 2500 copies.

Papers on Soils (STP 206), 120 pp., 2000 copies.

Symposium on Industrial Water and Industrial Waste Water (STP 207), 56 pp., 2500 copies.

Symposium on Radiation Effects on Materials (STP 208), 196 pp., 3000 copies.

Design and Test of Building Structures—Seismic and Shock Loading, Glued Laminated and Other Wood Constructions (STP 209), 90 pp., 2500 copies.

Symposium on Full-Scale Tests on House Structures (STP 210), 64 pp., 2500 copies.

Symposium on Steam Turbine Oils (STP 211), 110 pp., 2500 copies.

Symposium on Aircraft Fatigue (STP 203), 48 pp., 2000 copies.

Papers on Road and Paving Materials (STP 212), 82 pp., 2000 copies.

Symposium on Railroad Materials (Including Symposium on Lubricating Oils) (STP 214), 180 pp., 2500 copies.

Symposium on Nondestructive Testing (STP 213), 96 pp., 2500 copies.

Symposium on Radioisotopes (STP 215), 120 pp., 2500 copies.

Symposium on Thermal Conductivity Measurements and Applications of Thermal Insulation (STP 217), 92 pp., 2500 copies.

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STP numbers, page size and quantity not yet assigned for the following publications:

Symposium on Nondestructive Tests in the Nuclear Energy Field

Symposium on Composition of Petroleum Oils

Data on Knock Test Characteristics

Respectfully submitted on behalf of the committee,

R. E. HESS,
*Chairman*

## APPENDIX V

# REPORT OF ADMINISTRATIVE COMMITTEE ON END-USE PRODUCTS

Since the last report of the Administrative Committee on Ultimate Consumer Goods, which appeared in the ASTM *Proceedings*, Vol. 51 (1951), the following interim events are reported as a matter of record.

At the Annual Meeting of the Society in June, 1951, the Administrative Committee sponsored a Symposium on Measurement of Consumer Wants for the purpose of presenting to the members of the Society some of the procedures which have been developed for the evaluation of an individual's wants and preferences. The papers and authors were as follows:

"Determination of Soldier Wants" by W. C. Schaefer.
"Determination of Soldiers' Food Wants" by R. Palmer Benedict.
"The General Problem of Measurement" by Samuel A. Stouffer.
"Interviewer Bias" by Clyde Hart.
"Some Application of the Panel Technique in Social Resarch" by Charles Y. Glock.
"Effective Sampling Procedures" by F. F. Stephan and P. J. McCarthy.

These have been issued by the Society as *STP No. 117.*

A meeting of the Administrative Committee was held on November 2, 1955, for the purpose of examining critically the usefulness of the committee to the work of the Society. The discussion resulted in recommendations to the Board of Directors for a change in title and scope of the committee as follows:

> *Title:* Administrative Committee on End-Use Products.
>
> *Scope:* (1) To promote knowledge of engineering test methods for end-use products; (2) To encourage and stimulate investigations for the above purpose, both from the engineering point of view and from that of the fact-finder seeking to determine the needs and wants of the user of end-products; (3) To advise with and assist technical committees which may desire to establish measures or scales of performance, or standards, for end-use products based on their test methods; (4) To review annually for the Society progress in the field of testing of end-use products and in the development of standards for such products.

These changes were approved by the Directors at a meeting on May 8, 1956.

A further meeting of the Administrative Committee was held on November 15, 1956, devoted in large part to an exploration of the field of sensory testing, particularly for appearance, odor or taste, and the extent to which it has been developed in the work of the technical committees of the Society. M. Rea Paul described the work of Committee E-12 on Appearance. Amos Turk discussed odor measurements and the tests for odor which are found in standards sponsored by Committees C-16 on Thermal Insulating Materials, D-1 on Paints, D-17 on Naval Stores, D-19 on

Industrial Water, and D-22 on Methods of Atmospheric Sampling and Analysis. Dean Foster described some of the problems which arise in psychophysical testing and methods for reduction of error or bias. Paul S. Olmstead discussed the requisite for normality of response by panel judges, and the effect of sample size on reliability of results.

W. S. MacLeod presented the need of the General Services Administration for standards for end-products and indicated the extent to which it looked to ASTM for their development.

At the present time the committee has under consideration the development of a program of work.

Respectfully submitted on behalf of the committee,

HERBERT J. BALL,
*Chairman.*

FRANK Y. SPEIGHT,
*Secretary.*

## APPENDIX VI

# REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

Thirty-six local ASTM meetings were held during the past year, including 30 district meetings and 6 meetings held in areas not currently incorporated in any district. Meetings held in new areas included Jacksonville, Fla., Atlanta, Ga., Birmingham, Ala., and Madison, Wis. President R. A. Schatzel presented his district address "Materials for Electric Power Transmission" sixteen times. In addition he addressed four other meetings. Of the 36 meetings held, 19 were jointly sponsored with such societies as American Welding Society, National Association of Corrosion Engineers, American Society of Mechanical Engineers, American Society of Tool Engineers, American Chemical Society, and the Quality Concrete Conference held in Raleigh, N. C.

Dr. Charles E. Reed, the 1956 Marburg lecturer, presented his lecture "The Industrial Chemistry, Properties, and Applications of Silicones" in Los Angeles and San Francisco.

Many districts have initiated new approaches to the district meeting. The Chicago District sponsored an afternoon forum on the future outlook for ASTM. The New England District is continuing its program of sponsoring a spring meeting at one of the colleges or universities in its area. This year the meeting was held at the University of Rhode Island. Student Award Programs were held in the Philadelphia, Pittsburgh, Cleveland, Southwest, Northern California, Southern California, and Detroit Districts.

The Philadelphia District will be host for the 1957 Annual Meeting where it sponsors the Ladies' Entertainment and underwrites the entertainment following the Annual Dinner. An evening's entertainment was also sponsored by the Philadelphia District during the Annual Committee Week and Spring Meeting session held in Philadelphia, February, 1957.

Three of the 36 district meetings were held in conjunction with the formation of a district in the Southeast portion of the United States.

With the exception of the Ohio Valley District, at least one meeting was held in each District. More district meetings were held this year than at any time previous. In this connection, Headquarters, as has been the custom, prepared and distributed over 66,000 printed notices to ASTM members, committee members, and to cooperating societies in the particular districts as the events occurred. Council members in the 14 districts received all meeting notices. Of the 36 district meetings, 8 were Student Award Meetings.

The Northern and Southern California Districts cooperated in sponsoring the

Second Pacific Area National Meeting. The meeting was highlighted by a series of industry luncheons.

The Administrative Committee on District Activities held a meeting during the 1956 Annual Meeting and sponsored a breakfast meeting to which District Council officers were invited. The meeting served as an excellent means for extending ideas and information for the promotion of district activities.

Respectfully submitted on behalf of the committee,

C. M. GAMBRILL,
*Chairman.*

# REPORT OF COMMITTEE A-1
## ON
## STEEL*

As a result of increasing interest in spring steel wire, a new Subcommittee III on Spring Steel Wire was organized under the chairmanship of R. S. Thompson of the Washburn Wire Co. This necessitated changing the scope of the existing Subcommittee IV to cover hot formed spring steel and steel springs. Eight specifications for spring steel wire which had been prepared previously by Subcommittee IV and published by the Society will now be assigned to the new Subcommittee III. The first new specification proposed by Subcommittee III is attached to this report.[1]

Another development of widespread interest is the proposed Tentative Specifications for Special Large Size Deformed Billet Steel Concrete Reinforcement Bars.[1] A very definite need by the construction industry has been expressed for this product specification. This covers bars designated Nos. 14S and 18S which fall outside the scope of the Specifications for Billet-Steel Bars for Concrete Reinforcement (A 15 - 54 T)[2] and for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A 305 - 56 T).[3]

The ASA Chemical Industry Advisory Board several years ago recommended that ASTM develop a specification for electric-fusion-welded austenitic chromium-nickel alloy-steel pipe in nominal diameters of 14 to 30 in. and with wall thicknesses of schedules 5S and 10S. After many drafts were considered, one has finally been approved by Committee A-1 for reference to the Society for publication as tentative.[1]

*MEETINGS.*—Committee A-1 met on June 19, 1956, in Atlantic City, N. J., in conjunction with the 1956 Annual Meeting of the Society. On June 17 and 18 nineteen subcommittees, sections, and task groups convened to prepare proposals for committee action.

Approximately 225 Committee A-1 members met on January 21, 22, and 23, 1957 in Houston, Tex. Twenty subcommittees, sections, and task groups met on January 21 and 22 to prepare reports for the committee meeting on January 23. Attendance at the subcommittee meetings dealing with tubular products broke all recent records, with many visitors from the petroleum and chemical industries present.

*PERSONNEL.*—The committee consists of 314 voting members, including 144 producers, 128 consumers, and 42 general interest members.

As mentioned previously in this report, a new Subcommittee III on Spring Steel Wire has been organized with R. S. Thompson of the Washburn Wire Co. as chairman.

G. R. Locke of the Republic Steel Corp. has been appointed as vice-chairman and secretary of Subcommittee IX on Steel Tubing and Pipe to replace H. R. Redington, National Tube Co., who has retired. In Subcommittee XI on Steel for Boilers and Pressure Vessels, C. Floyd of the Babcock & Wilcox Co. has been appointed vice-chairman and secretary.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16-21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 1.

[2] 1955 Book of ASTM Standards, Part 1.

[3] 1956 Supplement to Book of ASTM Standards, Part 1.

Due to the death of E. O. Dixon, Ladish Co., the chairmanship of the Low-Temperature Advisory Group of Subcommittee XXII was left vacant. L. H. Carr, Edward Valves, Inc., has been appointed to fill the post formerly occupied by E. O. Dixon.

Since the reorganization of Subcommittee XIII on Methods of Testing in 1946, L. H. Winkler of Bethlehem Steel Co. has been acting as chairman. He has asked to be relieved of this assignment, and W. H. Mayo of the U. S. Steel Corp. has been appointed in his place.

The committee records, with great regret, the deaths of five outstanding Committee A-1 members—H. C. Boardman, E. O. Dixon, Shortridge Hardesty, A. E. White, and Karl D. Williams.

H. C. Boardman, Director of Research, Chicago Bridge and Iron Co., had represented his company on Committee A-1 for the past 14 years. He was particularly active in Subcommittee II (Structural Steel) and Subcommittee XI (Pressure Vessel Steel).

E. O. Dixon, Vice President in Charge of Research and Metallurgy, Ladish Co., had been active in ASTM standardization activities on steel products since 1937. He had served as chairman of the Low-Temperature Advisory Group of Subcommittee XXII since the organization of the group about ten years ago.

Shortridge Hardesty, an internationally famous bridge designer, had been an active member of Committee A-1 and Subcommittee II (Structural Steel) since 1943. For the past fifteen years K. D. Williams had ably represented the U. S. Dept. of Navy, Bureau of Ships on Committee A-1.

A. E. White, as a leader in the field of metallurgical research, made outstanding contributions to the metallurgy of metals for high-temperature service. His affiliation with Committee A-1 started in 1921. For many years he served as chairman of Subcommittee XXII on Valves, Flanges, Fittings, and Piping for High-Temperature and Subatmospheric Temperature Service. The committee elected him to honorary membership in 1953. Dr. White was ASTM President in 1937–1938. In 1956 he was accorded the Society's highest honor, an ASTM honorary membership.

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On November 21, 1956, the Administrative Committee on Standards Acting for the Society, accepted the recommendation of Committee A-1 to withdraw the following Emergency Alternate Provisions:

*Emergency Alternate Provisions:*

| | |
|---|---|
| EA - A269a. | Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 56), |
| EA - A271a. | Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 56), |
| EA - A312a. | Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 56 T), and |
| EA - A329a. | Spec. for Heat-Treated Steel Tires (A 329 - 54). |

## RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 5 new tentatives and is recommending the revision of 24 tentatives and 15 standards and the adoption as standard of 1 tentative. In addition a tentative revision of one standard is being recommended.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.[4]

The recommendations in the report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

## ACTIVITIES OF SUBCOMMITTEES

The highlights of many current subcommittee activities are described in the following paragraphs:

Subcommittee V on Steel Reinforcement Bars has become very active. Proposed specifications for hot-rolled plain rods in coils and also for hot-rolled deformed rods in coils are being prepared. A third proposed specification covers uncoated seven wire stress-relieved strand for prestressed concrete reinforcement.

In tubular products, specifications for seamless and electric resistance welded low-alloy steel tubes for corrosion resistance and for middle welded single length still tubes are in near final form. Also specifications for centrifugally cast ferritic alloy steel pipe for high-temperature service are being prepared. A proposal has been made to approve the inclusion of the LD steel process (basic oxygen process) in the Specifications for Black and Hot-Dipped Zinc Coated Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 54).

Subcommittee XIX on Steel Sheets and Sheet Steel has numerous projects under way. These include proposed specifications for hot-rolled carbon steel sheets, commercial quality, for carbon steel sheets of flange and firebox qualities, for enameling iron sheets, and for low-alloy high-tensile steel sheets of commercial quality.

Subcommittee XXVII on Steel Chain has reached almost final agreement on proposed specifications for carbon-steel chain of three strength levels. As reported last year, this specification is a result of the withdrawal of requirements for carbon steel chain from Specifications A 56 – 39.

This report has been submitted to letter ballot of the committee, which consists of 314 members; 212 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. F. COLLINS,
*Chairman.*

H. L. FRY,
*Secretary.*

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[4] See p. 85.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

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## EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

*New Tentative Specifications for:*

General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy-Steel Bars (A 29 – 57 T).

*Revision of Tentative Specifications for:*

General Requirements for Delivery of Rolled Structural Steel (A 6 – 56 T),
Hot-Rolled Carbon-Steel Bars (A 107 – 52a T),
Cold-Finished Carbon-Steel Bars and Shafting (A 108 – 52 T),

Heat-Treated Alloy-Steel Bars (A 286 – 52 T),
Alloy-Steel Bars to End-Quench Hardenability Requirements (A 304 – 55 T),
Carbon-Steel Bars Subject to Mechanical Property Requirements (A 306 – 55 T),
Stress-Relief-Annealed Cold-Drawn Carbon-Steel Bars (A 311 – 52 T),
Heat-Treated Carbon-Steel Bars (A 321 – 52 T),
Hot-Rolled Alloy-Steel Bars (A 322 – 52 T),
Cold-Finished Alloy-Steel Bars (A 331 – 50 T),
Alloy-Steel Bars for Nitriding (A 355 – 52 T), and
Cold-Finished Heat-Treated Alloy-Steel Bars (A 364 – 52 T).

The new and revised tentative specifications appear in the 1957 Supplement to Book of ASTM Standards, Part 1.

On September 15, 1957, the Administrative Committee on Standards accepted the following recommendations which appear in Part 1 of the 1957 Supplement:

*New Tentative Specifications for:*

Carbon Steel Chain (A 413 – 57 T),
Carbon Steel Sheets of Flange and Firebox Qualities (A 414 – 57 T),
Hot-Rolled Carbon Steel Sheets, Commercial Quality (A 415 – 57 T),
Uncoated Seven-Wire Stress-Relieved Strand for Prestressed Concrete (A 416 – 57 T), and
Upholstery Spring Wire for Zig-Zag Type and Square Formed Type and No-Sag Type Spring Units (A 417 – 57 T).

*New Tentative Method for:*

Ultrasonic Testing and Inspection of Turbine and Generator Steel Rotor Forgings (A 418 – 57 T).

*Revision of Tentative Specifications for:*

Carbon-Steel Axles for Cars and Tenders (A 21 – 57 T),
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 56 T),
Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 56 T),
Heavy Gage Structural-Quality Flat Hot-Rolled Carbon-Steel Sheets (A 245 – 52 T),
Carbon and Alloy-Steel Forgings for Turbine Rotors and Shafts (A 293 – 55 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 56 T),
Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 55 T),
Alloy-Steel Seamless Drum Forgings (A 336 – 57 T),
Ferritic and Austenitic Steel Castings for High Temperature Service (A 351 – 52 T),
Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 55 T),
Cold-Rolled Carbon-Steel Deep-Drawing Sheets, Special Killed for Miscellaneous Drawn or Severely Formed Parts (A 365 – 53 T),
Cold-Rolled Carbon-Steel Sheets, Commercial Quality (A 366 – 53 T), and
Alloy-Steel Castings Specially Heat Treated for Pressure Containing Parts Suitable for High-Temperature Service (A 389 – 56 T).

*Revision and Reversion to Tentative of Standard Specifications for:*

Black and Hot-Dipped Zinc Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 54),
Alloy-Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 55),
Carbon-Steel Forgings for Railway Use (A 236 – 57),
Carbon and Alloy-Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288 – 55),
Carbon and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 55),
Carbon and Alloy-Steel Forgings for Turbine Bucket Wheels (A 294 – 55), and
Forged or Rolled Carbon and Alloy-Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 – 57),

## APPENDIX

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## PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix recommendations are given affecting certain specifications both standard and tentative, covering various steel products. The specifications appear in their present form in either the 1955 Book of ASTM Standards, Part 1, or the 1956 Supplement to Book of ASTM Standards, Part 1.

### NEW TENTATIVES

The committee recommends for publication as tentative the following specifications as appended hereto:[1]

*Tentative Specifications for:*

High Strength Structural Alloy Rivet Steel,
Upholstery Spring Wire for Coiled Type Springs,
Special Large Size Deformed Billet Steel Concrete Reinforcement Bars,
Welded Large Outside Diameter Light Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service, and
Chromium-Copper-Nickel-Aluminum Alloy Steel Plates for Pressure Vessels.

### REVISIONS OF TENTATIVE

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

**Tentative Specifications for Billet-Steel Bars for Concrete Reinforcement (A 15 – 56 T):**

*Table V.*—Revise footnote *a* to read, "The term 'lot' means all bars of the same nominal weight per linear foot contained in an individual shipping release or shipping order."

**Tentative Specifications for Rail-Steel Bars for Concrete Reinforcement (A 16 – 54 T):**

*Table V.*—Revise as indicated above for Table V of Specifications A 15.

**Tentative Specification for Carbon-Steel Axles for Cars and Tenders (A 21 – 55 T):**

*Section 1.*—Revise Note 3 to read, "Sections 12, 13 (*a*), 13 (*b*), 14, 15, 16, 17, and 18(*b*) of this specification, covering permissible variations, workmanship, finish, weights, marking, storing, and inspection, are applicable to heat treated axles corresponding to the size classification shown in Table II ordered in accordance with Specification A 236.[2]"

*Section 12.*—Revise to read as follows:

12. (*a*) *Length:*

(*1*) For plain bearing axles, the overall length shall not be less than the specified minimum length nor more than $\frac{1}{8}$ in. over.

(*2*) For passenger car roller bearing axles ordered finished to length, the overall length shall not be less than the specified minimum length nor more than $\frac{1}{16}$ in. over.

(*3*) For all axles ordered for finished end facing by the purchaser, the overall length shall range from $\frac{1}{8}$ to $\frac{1}{4}$ in. over the specified minimum length.

(*4*) For any other axles ordered to final length and not covered by the above classifications, the overall length shall not be less than the specified minimum length nor more than $\frac{1}{8}$ in. over.

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[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 1.

[2] 1955 Book of ASTM Standards, Part 1.

NOTE.—The specified minimum length shall be the nominal overall final length less any allowable tolerance.

(*b*) *Axles with Rough Turned Journal and Wheel Seat Portions and Smooth Forged Bodies.*—The diameters of the straight center portion and the intermediate tapered portion of the axles shall not be under the dimensions specified and shall be forged in a workmanlike manner. For proper finish machining of the journal and wheel seat portions, there shall be allowed $\frac{1}{8}$ to $\frac{1}{4}$ in. of metal over the finish sizes on diameters and longitudinally at each change of cross-section.

(*c*) *Axles with Rough Turned Journal and Wheel Seat Portions and Turned Bodies:*

(*1*) The rough turning of journal and wheel seat portions shall be as specified in Paragraph (*b*).

(*2*) The straight center portion and the intermediate tapered portion of the axles, when turned to specified size, shall not be more than $\frac{1}{8}$ in. over on diameters and shall have not more than $\frac{1}{8}$ in. metal allowed longitudinally at each change of cross-section.

(*3*) The center portion, when rough turned with an allowance for finish, shall be $\frac{1}{8}$ to $\frac{1}{4}$ in. over the finish size on diameter and shall have $\frac{1}{8}$ to $\frac{1}{4}$ in. metal allowed longitudinally at each change of cross-section with the following exceptions: On standard passenger car axles and alternate standard roller bearing freight car axles, the dimension between the inner edges of the wheel seat shall be to size with no more than $\frac{1}{8}$ in. metal allowed at each change of cross-section.

(*4*) Machining shall be done in a workmanlike manner to contour specified.

### Tentative Specifications for Welded and Seamless Steel Pipe (A 53 – 55 T):

*Section 7* (*c*).—Add a new second sentence to read, "For electric-resistance-welded pipe produced in multiple lengths, flattening tests are required only on the crop ends from the front and back of each coil."

### Tentative Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 55 T):

*Section 4* (*a*).—Revise to read, "Material for forgings shall consist of ingots or blooms, billets, slabs, or bars, either forged or rolled from an ingot, and cut to the required length by a suitable process."

*Section 12.*—Revise to read as follows:

12. If any of the results of the mechanical tests of any test lot do not conform to the requirements specified, the manufacturer may retest such lots but not more than twice, except with the approval of the purchaser on the basis of satisfactory metallurgical evidence that the cause of the failure is curable and the quality of the material is satisfactory. Retests shall be made as specified in Section 11 (*a*).

### Tentative Specifications for Electric-Resistance-Welded Steel Pipe (A 135 – 55 T):

*Section 7.*—Add a new second sentence as indicated above for Section 7 (*c*) of Specifications A 53.

### Tentative Specifications for Axle-Steel Bars for Concrete Reinforcement (A 160 – 54 T):

*Table V.*—Revise as indicated above for Table V of Specifications A 15.

### Tentative Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 – 55 T):

*Section 4* (*a*).—Revise as indicated above for Section 4 (*a*) of Specifications A 105.

*Section 11.*—Revise as indicated above for Section 12 of Specifications A 105, referring to Section 10 (*a*) in the last sentence.

### Tentative Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 56 T):

*Section 4* (*a*).—Revise as indicated above for Section 4 (*a*) of Specifications A 105.

*Section 10.*—Revise to read as follows:

10. *Hydrostatic Tests.*—(*a*) Valve bodies or fittings and other pressure-containing parts shall

be tested after machining to the hydrostatic shell test pressures prescribed in the American Standard for Steel Pipe Flanges and Flanged Fittings (ASA No. B16.5-1953) for the applicable alloy steel rating for which the forging is designed and shall show no leaks. Forgings ordered under these specifications for working pressures other than those listed in the American Standard ratings shall be tested to such pressures as may be agreed upon by the manufacturer and the purchaser.

(*b*) No hydrostatic test is required for welding neck or other flanges.

(*c*) It is realized that the forging manufacturer cannot perform pressure tests on rough forgings which are to be finally machined by others. Also, the fabricator of some finished forged parts cannot always test forgings which are designed to be pressure containing only after assembly by welding into a larger structure. However, the manufacturer of such forgings is responsible as required under Section 19(*b*) for the satisfactory performance of the forgings under the final test required in Paragraph (*a*).

*Section 13.*—Revise to read as indicated above for Section 12 of Specifications A 105, except refer to Section 12 (*a*) in the last sentence.

### Tentative Specifications for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 55 T):

*Section 1* (*a*).—Delete the last two sentences, which removes wall thickness limitations from the specifications.

### Tentative Specification for Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 54 T):

*Table I.*—Add a footnote to read, "When specified, the maximum incidental copper content shall be 0.25 per cent." Add an additional footnote to read "When specified, the steel shall be coarse grained having a carburized austenite grain size of 1 to 5 as determined in accordance with the Classification of Austenite Grain Size in Steel (ASTM Designation: E 19)[2] by carburizing at 1700 F for 8 hr."

### Tentative Specification for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 212 – 54a T):

*Table I.*—Add two new footnotes as indicated above for Table I of Specifications A 201.

TABLE I.—CHEMICAL REQUIREMENTS.

(Additional to Table II, Specifications A 213 – 56 T, Table I, Specifications A 249 – 56 T, Table I, Specifications A 269 – 56, and Table I, Specifications A 312 – 56 T).

| | Grade TP 304L | Grade TP 316L |
|---|---|---|
| Carbon, max, per cent..... | 0.035[a] | 0.035[a] |
| Manganese, max, per cent.. | 2.00 | 2.00 |
| Phosphorus, max, per cent... | 0.040 | 0.040* |
| Sulfur, max, per cent...... | 0.030 | 0.030 |
| Silicon, max, per cent...... | 0.75 | 0.75 |
| Nickel, per cent........... | 8.00 to 13.00 | 10.00 to 15.00 |
| Chromium, per cent....... | 18.00 to 20.00 | 16.00 to 18.00 |
| Molybdenum, per cent..... | .... | 2.00 to 3.00 |

[a] For small diameter or thin walls, or both, where many drawing passes are required, a carbon maximum of 0.040 per cent is necessary in grades TP 304L and TP 316L.

* 0.030 per cent, max, for Specifications A 213 only.

### Tentative Specifications for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 – 56 T):

*Table II.*—Add grades TP 304L and TP 316L as shown in the accompanying Table I, except that for grade TP 316L the maximum phosphorus content shall be 0.030 per cent. For grades TP 304, TP 310, and TP 348 change the maximum phosphorus content from "0.030" to read "0.040" per cent.

*Table III.*—Change the table to require for the new grades TP 304L and TP 316L a minimum yield point of 25,000 psi, a minimum tensile strength of 70,000 psi, and a minimum elongation in 2 in. of 30 per cent.

### Tentative Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 56 T):

*Section 1 (a).*—Revise as indicated above for Section 1 (*a*) of Specifications A 192.

### Tentative Specifications for Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 – 56 T):

*Table I.*—Add the following footnote referenced to grade WPC fittings, "No ASTM specifications have as yet been written for plate or forgings of this grade. However fittings may be made from plate or forgings which conform in mechanical and chemical properties to grade C of Specifications A 106."

### Tentative Specifications for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 – 56 T):

*Table I.*—Add grades TP 304L and TP 316L as shown in the accompanying Table I. Add grade TP 309 with chemical requirements as follows:

| | |
|---|---|
| Carbon, max, per cent | 0.15 |
| Manganese, max, per cent | 2.00 |
| Phosphorus, max, per cent | 0.040 |
| Sulfur, max, per cent | 0.030 |
| Silicon, max, per cent | 0.75 |
| Nickel, per cent | 12.0 to 15.0 |
| Chromium, per cent | 22.0 to 24.0 |

For all grades change the maximum phosphorus content from "0.030" to read "0.040" per cent.

*Table II.*—Revise as indicated above for Table III in Specifications A 213.

### Tentative Specification for Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 285 – 54 T):

*Table I.*—Add a new footnote to read, "When specified, the maximum incidental copper content shall be 0.25 per cent."

### Tentative Specification for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 56 T):

*Table I.*—Add grades TP 304L and TP 316L as shown in the accompanying Table I. For all grades change the maximum phosphorus content from the present "0.030" to read "0.040" per cent.

*Table II.*—Revise as indicated above for Table III of Specifications A 213.

### Tentative Specification for Alloy Steel Seamless Drum Forgings (A 336 – 55 T):

*Section 7 (c).*—Revise the first sentence to read, "The ferritic steels shall be either fully annealed or normalized and tempered."

### Tentative Specifications for Heavy-Walled Carbon and Low Alloy Steel Castings for Steam Turbines (A 356 – 56 T):

*Section 7 (b).*—Revise to read as follows:

> (*b*) The yield point shall be determined by the drop of the beam or the halt in the gage of the testing machine, by the use of dividers, or by other approved methods. When a definite yield point is not exhibited, the yield strength corresponding to a permanent offset of 0.2 per cent of the gage length of the specimen, or to a total extension under load of 0.5 per cent of the gage length shall be determined, or the yield strength determined by any other means mutually agreeable to the manufacturer and the purchaser may be substituted.

*New Section.*—Add a new Section 9 on Test Specimens and Methods of Tension Testing to read as follows, renumbering the present Section 9 and subsequent sections accordingly:

> 9. The test specimens and the tensile tests required by these specifications shall conform to those described in the Tentative Methods and

TABLE II.—PERMISSIBLE VARIATIONS IN CHECK ANALYSIS.
(New Table II, Specification A 383 - 55 T)

| Element | Permissible Variation, per cent | |
|---|---|---|
| | Over | Under |
| Manganese | 0.03 | 0.03 |
| Phosphorus | 0.008 | .... |
| Sulfur | 0.008 | .... |
| Silicon | .... | 0.02 |

Definitions for the Mechanical Testing of Steel Products (ASTM Designation: A 370).[2]

*Section 8.*—Delete Paragraph (*c*).

*Figure 2.*—Delete this figure.

### Tentative Specification for Untreated Carbon Steel Axles for Export and for General Industrial Use (A 383 - 55 T):

*Section 8.*—Revise the last sentence to read, "The chemical composition thus determined shall not vary from the requirements specified in Table I by more than the amounts prescribed in Table II."

*New Table.*—Add a new Table II to read as shown in the accompanying Table II, renumbering subsequent tables accordingly.

*Section 17.*—Reletter the present text as Paragraph (*a*) and add a new Paragraph (*b*) to read as follows:

> (*b*) The inspector shall examine each axle in each heat for workmanship, defects, and conformity to the dimensions given on the order or drawing. If in this inspection defects are found which the manufacturer can remedy while the inspector is at the works, he may be allowed to correct such defects.

### Tentative Specification for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 387 - 56 T):

*Table I.*—For grade B in group I revise the check analysis requirements for chromium from "0.75 to 1.21" to read "0.74 to 1.21" per cent.

*Section 9 (b).*—Delete the words "in group I."

*Section 11 (c).*—Revise to read as follows:

> (*c*) Tension test specimens for group II plates for thicknesses under $\frac{3}{4}$ in. shall conform to the dimensions shown in Fig. 5 (or alternatively Fig. 4) of Methods A 370, except if desired, they may have straight or parallel edges (dimensions: plate thickness by $\frac{3}{4} \pm 0.015$ in. width by 8 in. minimum length). On plates $\frac{3}{4}$ in. and over in thickness, the group II tension test specimens shall be machined to the form and dimensions of Fig. 6 of Methods A 370.

### Tentative Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts Specially Heat-Treated for High-Temperature Service (A 404 - 56 T):

*Section 10.*—Revise as indicated above for Section 10 of Specifications A 182.

## TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as follows of the Standard Specifications for Hard-Drawn Steel Spring Wire (**A 227 - 47**):

*Title.*—Revise to read, "Hard-Drawn Steel Mechanical Spring Wire."

*Section 1.*—Revise to read, "These specifications cover a grade of round steel wire known as hard-drawn mechanical spring wire, uniform in quality, intended especially for the manufacture of mechanical springs."

## ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specification for High-Strength Structural Rivet Steel (**A 195 - 52 T**) be approved for reference to letter ballot of the Society for adoption as standard without revision.

## REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual

Meeting in order that the revision may be referred to letter ballot of the Society:

### Standard Specifications for Wrought Steel Wheels for Electric Railway Service (A 25 – 48):

*Section 4.*—Delete footnote *a* from the table of chemical requirements.

*Figure 1.*—Remove the lower arrow head for dimension "d."

*Section 10.*—Revise the second sentence of paragraph (*a*) to read, "The dies used to produce characters shall be $\frac{1}{2}$ in. in nominal height at the crest and hot stamping shall be nominally $\frac{3}{32}$ in. in depth." Delete Paragraph (*c*).

*Appendix.*—Delete the appendix.

### Standard Specifications for Steel Tires (A 26 – 39):

*Section 2* (*b*).—Revise to read "All tires, immediately after being rolled, shall be slowly cooled or handled in a manner so as to prevent injury by too rapid cooling below the critical range."

*Section 4.*—Delete the requirements for nickel and chromium content.

*Section 5.*—Change the words "test ingot" to read "test specimen."

*Section 7.*—Revise to read, "Tension tests from representative bars made in accordance with Section 8 shall conform to the following requirements as to tensile properties:"

*Figure 1.*—Replace with Fig. 6 of the Tentative Methods and Definitions for Mechanical Testing of Steel Products (ASTM Designation: A 370).[2]

*Section 9.*—Revise to read as follows:

9. *Number of Tests.*—(*a*) One tension test shall be made from each heat.

(*b*) If any test specimen fails because of mechanical reasons, such as testing equipment failure, etc., it may be discarded and another specimen substituted.

*Section 10.*—Revise to read as follows:

10. *Retests.*—(*a*) If the results of the mechanical tests of any test lot do not conform to the requirements specified because a flaw develops in the test specimen during testing, a retest of a test specimen cut from the test ingot shall be allowed if the defect is not caused by ruptures, cracks, or flakes in the steel. If the defect is caused by ruptures, cracks, or flakes in the steel, a retest shall be allowed of a specimen cut from a tire as provided in Paragraph (*c*).

(*b*) If the percentage of elongation of any test specimen is less than that specified in Section 7 and any part of the fracture is outside the middle half of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

(*c*) If the results of the tension test for any heat do not conform to the requirements of Section 7, a retest may be made on a specimen cut from a tire of the same heat furnished at the expense of the manufacturer. This retest shall give results conforming to the requirements of Section 7. In case such a retest is made, the tension test specimen shall be located as shown in Fig. 2.

*New Figure.*—Add a new Fig. 2 to be the same as the present Fig. 1 of the Standard Specifications for Heat-Treated Steel Tires (A 329 – 54).[2]

*Section 14.*—Revise to read as follows:

14. *Marking.*—(*a*) The name or brand of the manufacturer, serial number, heat number, designation of class (A, B, or C) and date of manufacture shall be legibly stamped on each tire approximately $\frac{1}{4}$ in. from the inner edge where they will not be removed by the railroad's last turning. Cold stamping will be permitted on locomotive tires only when the hot stamping has been removed due to machining. The height of the characters shall not be less than $\frac{1}{2}$ in. for hot or cold stamping.

(*b*) The set number shall be legibly stenciled with paint on each tire.

*Section 15* (*a*).—Revise the last sentence to read, "Tests and inspection shall be made at the place of manufacture prior to shipment unless otherwise specified."

*Supplementary Requirement.*—Add a supplementary requirement to these specifications to read as follows, with the title "Incidental Alloy Content:"

S1. The purchaser may specify maximum amounts of incidental alloying elements as follows:

| | |
|---|---|
| Chromium, max, per cent | 0.15 |
| Nickel, max, per cent | 0.25 |
| Molybdenum, max, per cent | 0.06 |

### Standard Specifications for Mild-to Medium-Strength Carbon-Steel Castings for General Applications (A 27 – 55):

*Section 8 (b).*—Revise as indicated above for Section 7 (*b*) of Tentative Specifications A 356.

*Section 9.*—Retitle "Test Specimens and Methods of Tension Testing" and revise Paragraph (*b*) to read the same as new Section 9 of Tentative Specifications A 356.

*Figure 1.*—Delete this figure.

### Standard Specifications for Multiple-Wear Wrought Steel Wheels (A 57 – 54):

*Section 13 (q).*—Revise to read as follows:

(*q*) Within the above limitations on length of hub and depression of hub, the distance from the back face of the rim to the back face of the hub shall not vary more than $\frac{1}{8}$ in. over or under that specified. When hub faces are to be finish machined, extra stock of $\frac{1}{16}$ to $\frac{3}{16}$ in. per face may be left for finish or the hub face may be furnished finish machined, as specified. The hub length tolerance in this case is based on finish dimensions.

*Section 15 (a).*—Revise as indicated above for Section 10 (*a*) of Standard Specifications A 25.

### Standard Specifications for High-Strength Steel Castings for Structural Purposes (A 148 – 55):

*Section 8 (b).*—Revise as indicated above for Section 7 (*b*) of Tentative Specifications A 356.

*Section 9.*—Retitle "Test Coupons" and delete Paragraph (*b*).

*New Section.*—Add a new Section 10 to read as indicated above for new Section 9 of Tentative Specifications A 356, renumbering subsequent sections accordingly.

*Figure 1.*—Delete this figure.

### Standard Specifications for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 56):

*Table I.*—Add a footnote to read, "When specified, the steel shall be coarse grained having a carburized austenite grain size of 1 to 5 as determined in accordance with the Classification of Austenite Grain Size in Steel (ASTM Designation: E 19) by carburizing at 1700 F for 8 hr."

### Standard Specification for Carbon Steel Forgings for Railway Use (A 236 – 55):

*Section 19.*—Letter the present text as Paragraph (*a*) and add a new Paragraph (*b*) to read as follows:

(*b*) The inspector shall examine each forging in each heat for workmanship, defects, and conformity to the dimensions given on the order or drawing. If in this inspection defects are found which the manufacturer can remedy while the inspector is at the works, he may be allowed to correct such defects.

### Standard Specification for Carbon Steel Seamless Drum Forgings (A 266 – 55):

*Section 7 (a).*—Add a new first sentence to read "All forgings shall receive a heat treatment."

### Standard Specifications for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 55):

*Table I.*—For all grades change the maximum phosphorus content from "0.030" to read "0.040" per cent.

### Standard Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 56):

*Table I.*—Add new grades TP 304L and TP 316L as shown in the accompanying Table I. For all other grades change the maximum phosphorus content from "0.030" to read "0.040" per cent.

### Standard Specifications for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 55):

*Table I.*—Change the maximum phosphorus content from "0.030" to read "0.040" per cent.

### Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 56):

*Table I.*—Change the maximum phosphorus content for all grades from "0.030" to read "0.040" per cent.

### Standard Specification for Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 – 56):

*Table I.*—Add a new class 5 referencing the Proposed Tentative Specification for Chromium-Copper-Nickel-Aluminum Alloy Steel Plates for Pressure Vessels[1] at a service temperature minimum test temperature of −150 F.

*Section 3 (c).*—Replace the first sentence with the following: "If the value for more than one specimen is below 15 ft lb, or if the value for one specimen is below 10 ft lb, a retest of three additional specimens shall be made, each of which must equal or exceed the specified minimum value of 15 ft lb. Such a retest shall be permitted only when the average value of the three specimens equals or exceeds 10 ft lb."

### Standard Specifications for Heat-Treated Steel Tires (A 329 – 54):

*Section 5 (a).*—Revise to read, "All tires, immediately after being rolled, shall be slowly cooled or handled in a manner so as to prevent injury by too rapid cooling below the critical range."

*Section 7.*—Revise the second sentence to read "This analysis shall be made from a test specimen taken during the pouring of the heat."

*Figure 2.*—Substitute for the present figure, Fig. 6 of the Tentative Methods and Definitions for Mechanical Testing of Steel Products (ASTM Designation: A 370).[2]

*Section 12 (b).*—Change the words "more than $\frac{3}{4}$ in. from the center of the gage length" to read "outside the middle half of the gage length."

*Section 16 (a).*—Revise to read, "The tires shall be grouped as to outside diameters and shipped in sets."

*Section 18.*—Letter the present text as Paragraph (*a*) and add a Paragraph (*b*) to read, "The set number shall be legibly stenciled with paint on each tire."

*Section 19.*—Revise the last sentence to read, "Tests and inspection shall be made at the place of manufacture prior to shipment unless otherwise specified."

*Supplementary Requirement S4.*—Delete this supplementary requirement.

### Standard Specifications for Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 – 55):

*Section 4 (a).*—Revise as indicated above for Section 4 (*a*) of Tentative Specifications A 105.

*Section 14 (a).*—Revise as indicated above for Section 12 of Tentative Specifications A 105, referring to Section 13 (*a*) in the last sentence.

# REPORT OF COMMITTEE A-3
## ON
# CAST IRON*

Committee A-3 on Cast Iron held two meetings during the year: in Atlantic City, N. J., on June 22, 1956, and in Philadelphia, Pa., on February 8, 1957. Many subcommittees met in conjunction with the main committee.

The committee consists of 137 members, of whom 135 are voting members; 45 are classified as producers, 52 as consumers, 38 as general interest, and 2 as consulting members.

The elevated temperature tests on cast iron are continuing at the Southern Research Inst. under the guidance of the Joint ASTM-ASME Committee on Effect of Temperature on the Properties of Metals. Thermal shock tests have been made on all of the materials. Test specimens are heated to 800 F in 90 sec and quenched with the cycle repeated until failure occurs. This work is scheduled for completion in 1957.

Committee A-3 has received information from the U. S. Atomic Energy Commission that the use of cast iron in nuclear reactors has been largely in connection with concrete for shielding certain facilities, and it is believed the present specifications for cast iron are adequate. Developments in the field of atomic energy are being followed in case such developments involve more extensive use of cast iron and make advisable the development of new specifications.

Committee A-3 is interested in the considerations of Committee E-12 on Appearance to prepare a Manual on appearance evaluation of materials. Appearance of castings is largely related to casting finish and the degree of surface roughness, and the proposed manual may have some application to this particular problem.

### AMERICAN STANDARDS

The following standards under the jurisdiction of Committee A-3 have been approved as American Standard by the American Standards Assn.:

*Standard Specifications for:*

Gray Iron Castings (**A 48 – 56; ASA G25.1—1956**), and

Lightweight and Thin-Sectioned Gray Iron Castings (**A 190 – 47; ASA G27.1—1956**).

### REVISION OF STANDARD
### IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions of the Standard Specifications for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F (**A 278 – 56**),[1] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

*Section 2(b).*—Change to read as follows:

(*b*) Castings intended for use above 450 F shall be stress relief annealed. The castings and test bars shall be stress relieved by placing them in a suitable furnace at a temperature not exceeding 400 F, heating them uniformly to the temperatures and for the times specified in Table I. The heating and cooling rates shall be uniform and not more than 400 F per hr for castings of 1 in. maximum metal section, nor more than 400 F divided by the maximum section thickness in inches for thicker castings.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1956 Supplement to Book of ASTM Standards, Part 1.

*New Table.*—Add a new Table I to read as shown in the accompanying Table I.

TABLE I.—STRESS RELIEVING REQUIREMENTS.

| Class | Metal Temperature, deg Fahr | Holding Time, hr[a] | |
|---|---|---|---|
| | | Minimum | Maximum |
| 40, 50, 60 | 1050 to 1200 | 2 | 12 |

[a] In no case shall the holding time be less than 1 hr per in. of maximum metal section, or in excess of 12 hr maximum, dependent upon which governs.

## ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specification for Cast Iron Pressure Pipe (**A 377 - 54 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee 6 on General Castings* (H. W. Lownie, Jr., chairman) considered revisions of the Specifications for Gray Iron Castings (A 48 - 56) consisting of reworking of all notes, correction of the problem when more than one ladle of iron is used to pour a single casting in case the material in one ladle does not meet the specifications, and revision of Fig. 2 requiring threaded ends on test specimens. Action on these revisions will be deferred until more important revisions may be required.

The joint task force with Subcommittee 11 on Methods of Testing is investigating a new cast iron specification employing test specimens which can be cast free from the shrinkage sometimes found in the present long cylindrical test bars specified in Specifications A 48. Specimens other than the present standard arbitration type used by various foundries are being investigated. It is hoped to develop test specimens which will not only yield bars of improved soundness but will also have the advantages of closer correlation between governing section size of casting and test bar, employing less metal for test specimens in the interest of economy, and promoting economy in the cost of machining and preparing test bars from the specimens.

*Subcommittee 12 on Austenitic Cast Iron* (Hugo Larson, chairman) is preparing a specification covering several commercial grades of austenitic cast iron.

*Subcommittee 22 on Elevated Temperature Properties of Cast Iron* (J. S. Vanick, chairman) prepared the revisions of Specifications A 278 referred to earlier in the report. Appendix notes are to be left unchanged until Subcommittee 6 sets a revision pattern in Specification A 48.

*Subcommittee 24 on Nodular Iron* (T. E. Eagan, chairman) is preparing a revision of Specification A 395 limiting the hardness of castings furnished to a maximum of 183 Brinell hardness.

*Subcommittee 26 on Low Temperature Properties of Cast Iron* (George Dinges, chairman) is considering a draft of a recommended practice for the use of gray iron at low temperature.

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[2] 1955 Book of ASTM Standards, Part 1.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 135 voting members; 92 members returned their ballots, of whom 84 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. E. Krause,
*Chairman.*

R. A. Clark,
*Secretary.*

# REPORT OF COMMITTEE A-5

ON

# CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held two meetings during the year: on June 19, 1956, at Atlantic City, N. J., and on February 5, 1957, at Philadelphia, Pa. The committee consists of 107 members, of whom 50 are classified as producers, 33 as consumers, and 24 as general interest members.

The standards prepared by Committee A-5 have been issued in the form of a special Compilation of ASTM Standards on Zinc-Coated Iron and Steel Products.

A history of Committee A-5 is being prepared to commemorate the committee's 50th anniversary.

Various groups of corrosion reports sponsored by the committee are being assembled and brought up to date for publication. Each group will contain a complete story from the preparation of the original specimens to their last inspection.

In view of the fact that the Preece test has been removed from all specifications under the jurisdiction of the committee, an article explaining the shortcomings of this test was prepared by B. J. Barmack and appeared in the October, 1956 issue of the ASTM BULLETIN.

The advisability of Committee A-5 establishing a subcommittee on nuclear problems is under consideration.

## AMERICAN STANDARDS

The following standards under the jurisdiction of Committee A-5 were approved as American Standard by the American Standards Association:

*Specifications for:*

Zinc-Coated (Galvanized) Iron Telephone and Telegraph Line Wire (ASTM A 111 - 52; ASA C7.31—1956),

Zinc-Coated Steel Wire Strand (ASTM A 122 - 54 T; ASA C7.32—1956),

Zinc Coating (Hot-Dip) on Iron and Steel Hardware (ASTM A 153 - 53; ASA G8.14—1956),

Zinc-Coated Steel Wire Strand (ASTM A 218 - 54 T; ASA C7.33-1956),

Zinc-Coated (Galvanized) High Tensile Steel Telephone and Telegraph Line Wire (ASTM A 326 - 52; ASA C7.30—1956),

*Methods of Test for:*

Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles (ASTM A 90 - 53; ASA G8.12—1956), and

*Recommended Practice for:*

Safeguarding Against Embrittlement of Hot Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (ASTM A 143 - 46; ASA G8.13—1956).

The following specifications were reaffirmed as American Standard:

*Standard Specifications for:*

Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (ASTM A 116 - 48; ASA G8.9—1948), and

Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (ASTM A 121 - 48; ASA G8.10—1948).

## NEW TENTATIVE

The committee recommends that the Specification for Zinc-Coated (Galvanized) Low-Carbon Steel Armor Wire be accepted for publication as tentative as appended hereto.[1]

## REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] This specification appears in the 1957 Supplement to Book of ASTM Standards, Part 1.

*Standard Specifications for:*

Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (**A 116 - 48**):[2] Revise as appended hereto.[1]

Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (**A 121 - 48**):[2] Revise as appended hereto.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee VII Method of Testing* (O. B. Ellis, chairman) is investigating nondestructive tests for determining thickness of metallic coatings.

The Task Group on Magnetic Methods (V. V. Kendall, chairman) found that a magnetic gage method should be applicable to hot-dip galvanized, continuous galvanized, and galvannealed coatings, but the type of base metal may have an influence on the coating thickness indicated. Rimmed and killed steel showed considerable difference in thicknesses of zinc coatings when measured with a magnetic gage.

A large number of 4 by 6-in. steel panels with various coating thicknesses of zinc are being measured with magnetic instruments. Prior work had consisted of 1100 measurements on pipe specimens. Using a pocket-size gage having two polar pieces deviations from stripped weights were approximately 0.228 oz per sq ft. Results were evenly divided between plus and minus. Results may have been affected by residual magnetism in the base metal, which could be overcome by averaging readings taken by rotating the instrument 180 deg.

Because of the increasing use of aluminized steel products, a study has been undertaken of methods for the determination of weights and analysis of aluminum coatings on steel.

*Subcommittee XI on Sheet Specifications* (E. F. Lundeen, chairman) is considering changes in Specifications A 93 - 55 T, A 308 - 53 T, and A 361 - 55 T, and has therefore recommended that they be retained as tentative for the present.

A request that tinplate specifications be prepared has been tabled because changes taking place in this industry make it inadvisable to set up standards at the present time.

*Subcommittee XII on Wire Specifications* (J. F. Occasione, chairman).—The Section on Chain Link Fencing (B. A. Beery, chairman) is studying criticisms of recent tentative specifications. Deletion of the Preece test and other details have made it necessary to review the tentative specification for zinc-coated steel chain-link-fence fabric.

The section on telephone wire has proposed changes in Tentative Specification A 112 - 55 T for zinc-coated steel tie wires, subject to letter ballot. Further study is required with respect to proposed changes in Specifications A 111 - 52 and A 326 - 52.

The section on armor wire reports progress on a proposed tentative specification for flat armor tape.

The subcommittee prepared the revisions of the Standard Specifications for Zinc-Coated Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 - 48) and for Zinc-Coated Iron or Steel Barbed Wire (A 121 - 48) as appended to this report.

The subcommittee also prepared the proposed Tentative Specification for

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[2] 1955 Book of ASTM Standards, Part 1.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Zinc-Coated Low-Carbon Steel Armor Wire which is appended to this report.[1]

*Subcommittee XIII on Hardware Specifications* (B. J. Barmack, chairman).—This recommended practice for Safeguarding Against Embrittlement of Hot Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A 143 – 46) is being revised so as to emphasize precautions in shop practice regarding punching holes in heavy angles and the like. The advisability of raising the minimum weight of coating from the present 2 oz as defined in the Specification for Zinc (Hot-Galvanized) Coatings on Products Fabricated from Rolled, Pressed, and Forged Steel Shapes, Plates, Bars, and Strip (A 123 – 53) is being studied. Considerable test data for the Specification for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 – 53) are now being studied. Since the Preece test has been deleted from this specification, it has been suggested that a note be added covering the use of magnetic gages such as appears in Specification A 386 – 55 T.

*Subcommittee XIV on Sheet Tests* (C. P. Larrabee, chairman) makes detailed reports of its inspection activities in the even numbered years. The atmospheric tests of corrugated sheets at State College and Altoona, Pa., were inspected on April 24, 1957.

Color photographs were taken by this subcommittee for exhibit under the auspices of the Advisory Committee on Corrosion at both Atlantic City, N. J., and Los Angeles, Calif. in June and September, 1956, respectively.

*Subcommittee XV on Wire Tests* (F. M. Reinhart, chairman) inspected specimens of all sites where wire tests are in progress. A total of 436 wire specimens were removed for tension tests. A detailed report is appended.[4]

The 20-year contracts for the following wire test sites expired during 1956 and 10-year extensions are being negotiated by the Advisory Committee on Corrosion: Ames, Iowa; Manhattan, Kans.; College Station, Tex.; Davis, Calif.; Santa Cruz, Calif.; Ithaca, N. Y.; and Lafayette, Ind.

A summary paper is being prepared by this subcommittee covering the results of all wire exposure tests, starting with the original data and including the 1934 and 1939 wire test reports.

*Subcommittee XVI on Hardware Tests* (A. Mendizza, chairman) has added two forms of nodular iron to its test program. These will receive only a hot-dip zinc coating. Consideration is being given to the addition of malleable iron to the program.

The subcommittee is preparing 750 specimens for exposure early in 1958, at the following sites: Newark, N. J.; Kure Beach, 800-ft lot; and Kure Beach, 80-ft lot. Included in these tests are flat, bent, and round specimens made of carbon steel, several low-alloy steels, and nodular iron. These specimens are coated with hot-dip zinc, hot-dip aluminum, electrodeposited zinc, sprayed zinc, and sprayed aluminum.

A report of inspection of hardware specimens exposed at State College, Pa., is appended.[5]

This report has been submitted to letter ballot of the committee, which consisted of 107 members; 94 members returned their ballots, of whom 82 have voted affirmatively and none negatively.

Respectfully submitted on behalf of the committee,

MARC DARRIN,
*Chairman.*

C. P. LARRABEE,
*Secretary.*

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[4] See p. 99.

[5] See p. 130.

# REPORT OF SUBCOMMITTEE XV ON ATMOSPHERIC EXPOSURE TESTS OF WIRE AND WIRE PRODUCTS

## (COVERS 1955 AND 1956 INSPECTIONS)

This report summarizes the results of the atmospheric corrosion tests on wire and wire products through 1956 and presents in detail the data from 1955 and 1956 inspections. Reference to earlier reports of Committee A-5 on Corrosion of Iron and Steel which appeared in the Society's *Proceedings* for 1937, 1939, 1941, 1943, 1945, 1947, 1949, 1951, 1953, and 1955 will be helpful in studies of these data. These earlier reports contain descriptions of the test methods, micrographs, and other characterizations of specimens in the test as well as the data assembled during previous inspections.

The materials undergoing test are mostly bare and zinc-coated steel wires and wire products, but copper-covered and lead-coated steel wires and chromium and chromium-nickel steel wires are also included. The coatings are expressed in weight terms of ounces per square foot of surface. For comparisons of coating thickness, 1 oz per sq ft of surface may be considered as averaging 0.0017 in. thick for zinc coatings, 0.0013 in. thick for copper coatings, and 0.0010 in. thick for lead coatings.[1]

Specimens of wire and wire products have been exposed for about 20 yr at eleven sites. The locations of these sites and their types of atmosphere are as follows:

| Test Location | General Type of Atmosphere | Remarks |
|---|---|---|
| Pittsburgh, Pa. (abandoned) | Severe industrial | On Brunot Island in the Ohio River about 2 miles west of the city |
| Bridgeport, Conn. | Industrial | In the city |
| Sandy Hook, N. J. (abandoned) | Seacoast | About 300 yd from the Atlantic Ocean |
| Santa Cruz, Calif. | Rural (marine) | About 3 miles from the Pacific Ocean |
| State College, Pa. | Rural | Central Pennsylvania |
| Lafayette, Ind. | Rural | Wabash River Valley |
| Ames, Iowa | Rural | Central Iowa |
| Manhattan, Kans. | Rural | Kansas River Valley |
| Ithaca, N. Y. | Rural | Central New York |
| College Station, Tex. | Rural | About 120 miles inland |
| Davis, Calif. | Rural | About 80 miles inland |

The Pittsburgh, Pa., and Sandy Hook, N. J., sites were abandoned in 1951 and 1952, respectively.

At each location, more than 900 speci-

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[1] The lead-coated wires in these tests were lead coated over a bonding coat of zinc. See *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 87 (1943) for analysis of lead coatings.

TABLE A.—REPORT OF GROUP AVERAGES THROUGH 1956—20-YR EXPOSURE.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; MG = intermediate between "metallic" and "gray"; Y = yellowed or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes SR, SY = speckled appearance of rust, or yellowing; Br, Gn, respectively, signify corroded to a brown or green appearance; B = black; D = dark or dirt or soot excluding a better observation; GY = predominantly gray but showing indication of Y. Under each location the single center column designates the state of corrosion for those groups, which as a group, have not reached 1R or 100R;

| Coating Group or Weight of Zinc, oz per sq ft of surface | Number in Group | Bridgeport, Conn. | | State College, Pa. | | Lafayette, Ind. | | Ithaca, N. Y. | | Ames, Iowa | | Manhattan, Kans. | | College Station, Tex. | | Santa Cruz, Calif. | | Davis, Calif. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Average of Observed Years of Exposure Until 1R and 100R or State of Corrosion after Approximately 20-yr Exposure | | | | | | | | | | | | | | | | | |
| | | 1R | 100R | 1R | 100R | 1R | 100R | 1R | 100R | 1R | 100R | 1R | 100R | 1R | 100R | 1R | 100R | 1R | 100R |
| UNFABRICATED WIRES[a] | | | | | | | | | | | | | | | | | | | |
| 0.20 to 0.30 | 6 | 2.1 | 2.8 | 5.1 | 6.2 | 4.6 | 5.6 | 5.9 | 6.6 | 6.3 | 7.5 | 11.1 | 17.2 | 8.6 | 12.9 | 12.8 | 16.3 | 13.6 | 62R |
| 0.25 to 0.35 | 8 | 2.3 | 3.5 | 4.9 | 6.7 | 4.8 | 6.3 | 6.2 | 7.6 | 6.9 | 9.3 | 83R | | 8.9 | 91R | 84R | | 46R | |
| 0.35 to 0.45 | 7 | 2.9 | 3.7 | 6.6 | 8.4 | 5.5 | 6.8 | 7.4 | 8.5 | 8.4 | 11.4 | 32R | | 10.5 | 94R | 17.8 | 32R | Y | |
| 0.45 to 0.55 | 10 | 3.8 | 5.6 | 7.9 | 10.3 | 7.1 | 9.2 | 8.7 | 10.7 | 10.7 | 16.2 | 2R | | 11R | | Y | | Y | |
| 0.50 to 0.60 | 5 | 4.8 | 5.7 | 9.5 | 11.7 | 8.5 | 10.0 | 10.3 | 11.4 | 12.5 | 15.4 | Y | | 2R | | GY | | Y | |
| 0.60 to 0.70 | 12 | 5.4 | 7.1 | 10.2 | 13.2 | 8.8 | 11.4 | 10.9 | 13.1 | 79R | | Y | | 4R | | Y | | Y | |
| 0.70 to 0.85 | 10 | 5.7 | 7.1 | 11.3 | 14.3 | 9.9 | 12.1 | 11.6 | 13.8 | 15.8 | 75R | GY | | 1.5R | | G | | G | |
| 0.80 to 1.00 | 11 | 6.8 | 8.9 | 93R | | 11.2 | 15.0 | 13.6 | 16.4 | 22R | | GY | | 1R | | GY | | G | |
| 1.20 to 1.35 | 2 | 9.0 | 11.5 | 41R | | 16.0 | 84R | 18.3 | 56R | | Y | GY | | G | | ... | | ... | |
| 1.60 to 1.80 | 11 | 11.8 | 16.6 | 1R | | 3.9R | | 3.6R | | | G | G | | G | | G | | G | |
| 2.70 to 3.00 | 3 | | GY | G | | | G | G | | | G | G | | G | | G | | G | |
| Cu Coat | 2 | | Gn | Gn | | | Gn | Gn | | | Gn | B | | Br | | Br | | Br | |
| Pb Coat | 4 | Y | PHR | PHR | | 27 | PHR | 23R | | G | SR | 15R | | 7R | | 37R | | 20R | |
| 12–14% Cr | 6 | | GY | G | | | SR | B | | | SR | SR | | SR | | 65SR | | 46SR | |
| 18% Cr–8% Ni | 3 | | G | MG | | | M | M | | | MG | M | | MG | | MG | | MG | |

WIRE STRAND

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 0.69 to 0.93 | 4 | 9.9 15.3 | 2R | 15.0 23R | 17.1 25R | GY | G | G | G | G |
| 1.03 to 1.17 | 3 | 16.5 98R | 1R | 16R | 6R | GY | GY | G | G | G |
| 1.32 to 1.51 | 4 | 77R | GY | 4R | 3R | GY | GY | G | G | G |
| 1.71 to 2.47 | 2 | GY | G | G | G | G | G | G | G | G |
| Pb Coat | 1 | Y | PHR | PHR | 28R | Y SR | 23R | PHR | 68SR | 10R |

CHAIN LINK FENCE

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 0.29 to 0.52[b] | 3 | 2.9 4.8 | 6.4 9.1 | 5.7 9.0 | 5.9 11.5 | No Specimens Exposed | No Specimens Exposed | No Specimens Fxposed | 18.8 47R | 18.8 45R |
| 1.13 to 1.22[b] | 2 | 8.9 12.0 | 14.5 51R | 14.0 95R | 18.9 85R | | | | G | G |
| 2.09 to 2.92[b] | 2 | 5R | 1R | G | G | | | | G | G |
| 1.40 to 1.97[c] | 9 | 9.9 13.0 | 8R | 15.5 57R | 59R | | | | G | G |
| 4.21[c] | 1 | 10R | GY | GY | 19.9 8R | | | | MG | G |
| Cu Coat | 1 | Gn | Gn | Gn | Gn | | | | Br | Br |
| Pb Coat | 1 | Y | PHR | PHR | 23R | | | | 35SR | 20SR |
| 18% Cr-8% Ni | 1 | MG | M | MG | M | | | | MG | MG |

[a] Farm field fence and barbed wire made from these same wire lots corrode at about the same rate as the unfabricated wire, except that in the case of farm field fence the bottom part of the fence corrodes less then the upper part; in the case of barbed wire, the barbs show rust a year or two before rust appears on the main wire.

[b] Coated before weaving.

[c] Coated after weaving.

mens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations, farm field fence at nine sites, and barbed wire and chain link fence at eight sites.

The extent of corrosion is being measured by weight losses, by visual examinations of all specimens at the exposure sites, and by tension tests on wire specimens. The Bridgeport, Conn., State College, Pa., Sandy Hook, N. J., and Pittsburgh, Pa., specimens were inspected by a traveling committee. Inspections were made at the other locations by the university personnel in charge of the exposure plots.

*Findings from Weight Loss Specimens:*

At the start of the program, zinc-coated wire specimens were exposed at Pittsburgh, Pa., Sandy Hook, N. J., and State College, Pa., to determine the rate of loss of coating. These tests have now been completed, and results are summarized in the 1947 report. The over-all average annual loss of coating in ounces per square foot of surface was 0.369 at Pittsburgh, Pa.; 0.117 at Sandy Hook, N. J.; and 0.060 at State College, Pa.

*Findings from Visual Examinations for Corrosion at the Exposure Locations:*

The condensed and summarized inspection results of corrosion through 1956 are shown in Table A. In this table is shown the average number of years until first rust and until 100 per cent rust appeared on all specimens of each group where progress of rusting has been sufficient to determine the values at the various locations. For all other groups the average state of corrosion at the last inspection is shown. The galvanized specimens are divided into the coating weight groups established originally in the test to cover galvanized wire supplied under commercial specification at that time. However, determinations of the weights of coating of the wires, 1939 Report, showed that the coating weights of some wires were outside the range of their groups, i.e.; Wire No. 317 in the 0.25 to 0.35 group has a coating weight of 0.46 oz per sq ft of surface, Wire No. 235 in the 0.60 to 0.70 group has a coating weight of 0.82 oz per sq ft of surface, Wire No. 248 in the 0.80 to 1.00 group has a coating weight of 0.66 oz per sq ft of surface and Wire No. 450 in the 1.60 to 1.80 group has a coating weight of 1.48 oz per sq ft of surface. It is thought that placing these wires in the appropriate coating weight groups would reflect a clearer picture of the corrosion behavior of each group. This rearrangement will be made in the next report. The detailed inspection records collected in 1955 and 1956 are presented in Table I for unfabricated wire, Table II for barbed wire, Table III for farm field fence, Table IV for chain link fence, and Table V for wire strand. These tables are similar in form to the tables in previous reports.

The corrosion of any individual specimen of barbed wire or of farm field fence listed in Tables II and III may be directly compared with the performance of specific unfabricated wire specimens listed in Table I. The Reference Information Table shows the lot numbers of wires used in fabricating all farm field fence and barbed wire samples.

At the time of inspection, records are made of the appearance of each specimen. Prior to the time of rusting, the specimens are noted as appearing "metallic" (M), "gray" (G), "yellow" (Y), etc. After rusting has begun, estimates are made of the percentage of area rusted on each specimen. The term "rust" as given in the inspection instruction states: "Rust means real rust of the base metal—ordinarily a rough corroded area."

Because some of the exposure sites had never been visited by members of the subcommittee a resolution was passed at the 1952 Spring Meeting that a member visit and inspect these sites. In compliance with this resolution the sites at Ames, Iowa; Manhattan, Kans.; College Station, Tex.; and Davis and Santa Cruz, Calif., were visited between June 28 and July 27, 1956 to inspect the wires and wire products.

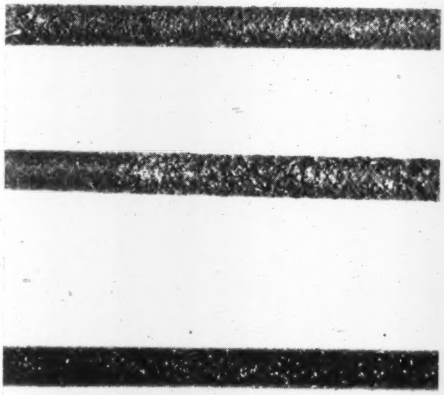

FIG. 1.—Wires Nos. 444, 443, and 441 Removed from Ames, Iowa, After 19.6 yr of Exposure.

Wire No. 444 shows typical appearance of the black, shiny, smooth condition of the wires at all locations. Light areas are pin points of light red rust. Wire No. 443 shows rough rust and a smooth area in center. Wire No. 441 is 100R (×1).

At these locations the hot dipped galvanized wires acquire a smooth, black shiny appearance where the zinc has weathered to the zinc-iron alloy layers instead of the "yellow" classification at the eastern sites. In some cases they were speckled with pin points of light red rust. According to the inspectors at the different sites this condition would last for varying periods of time; in some cases the wires will change to 100R within a year; in others discrete areas will turn to rough rust and gradually spread. Representative examples of this condition are shown in Fig. 1. The top wire is No. 444 (0.87 oz zinc per sq ft), smooth black coating with light red spots; the middle wire is No. 443 (0.74 oz zinc per sq ft), rough red rust except for smooth black area in center; the bottom wire is No. 441 (0.77 oz zinc per sq ft), 100R, removed from Ames, Iowa. Wire No. 444 is shown at higher magnification (X10) in Fig. 2. There is some justification for rating these wires as rusted even though they are smooth. The light red pin points in the black shiny coating certainly are rust spots and the brittle, glassy black coating conceivably could be ferric oxide. The warm dry weather with low relative humidity much of the time would be conducive to the formation of such a coating. An X-ray diffraction pattern of the powdered coating taken from one of the wires showed that it contained about 15 per cent alpha $Fe_2O_3 \cdot H_2O$ and 85 per cent amorphous powder.

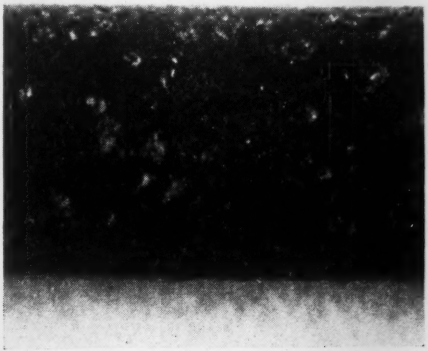

FIG. 2.—Wire No. 444, Shown in Fig. 1, at Higher Magnification to Show Light Red Spots in Black, Smooth Coating (×10).

The 12 to 14 per cent chromium steel wires had a mottled appearance with small spots of red rust scattered over their surfaces; typical examples are

FIG. 3.—Stainless Steel (12 to 14 per cent Cr) Wires Removed from College Station, Tex. after 19.6 yr of Exposure.

Top to bottom, cold-drawn, Nos. 001, 004, and 005; air quenched, Nos. 002, 006, and 007. Cold-drawn wires are more corroded than the air quenched (×1).

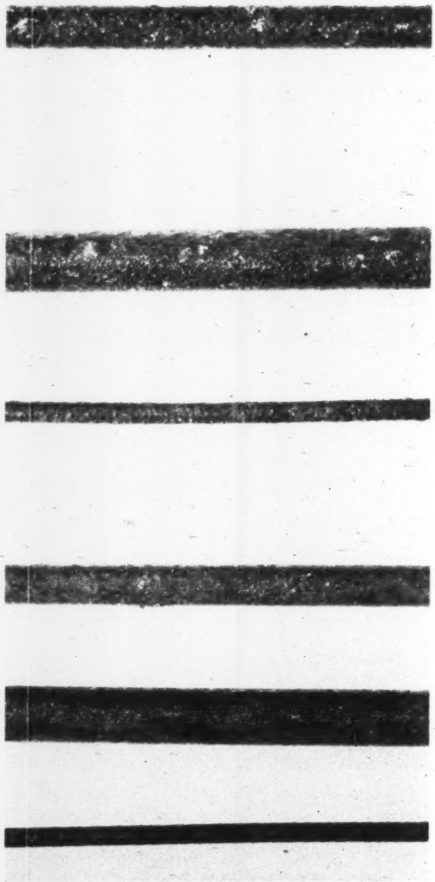

FIG. 4.—Stainless Steel (12 to 14 per cent Cr) Wires Removed from Davis, Calif., after 19.4 yr of Exposure.

Top to bottom, cold drawn, Nos. 001, 004, and 005; air quenched, Nos. 002, 006, and 007. Cold-drawn wires are more corroded than the air quenched. The wires at Davis, Calif., are more corroded than those at College Station, Tex. (×1).

shown in Figs. 3, 4 and 5. At all locations the cold drawn wires were more corroded than were the air quenched wires. These differences in behavior of the wires between the western and eastern exposure sites are probably due to the same climatic differences mentioned previously. The rainfall is not frequent enough to wash off the dust, which retains moisture from dew, forming local action cells.

All the unfabricated wires corroded faster on the undersides than on the topsides while the bottom portions of the chain link and farm field fence corroded either slightly faster or at about the same rate as the top portions, probably because of the retention of moisture on the lower portions longer than on the top portions.

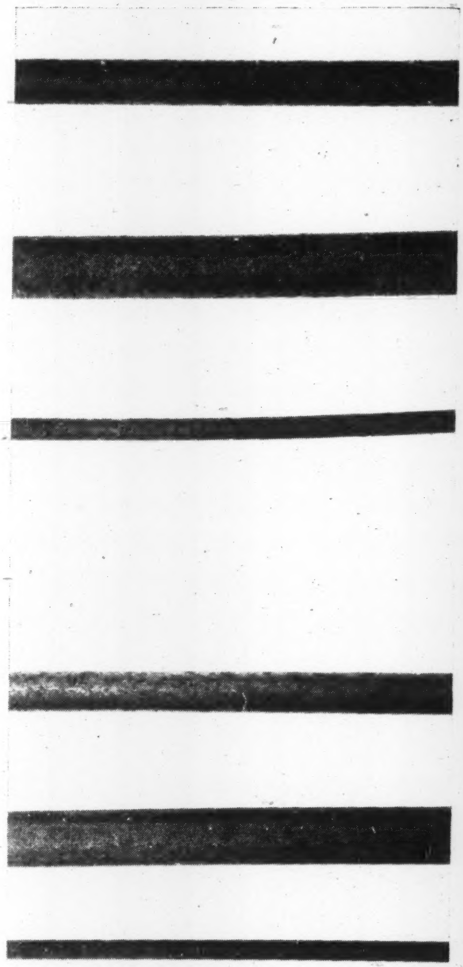

FIG. 5.—Stainless Steel (12 to 14 per cent Cr) Wires Removed from Santa Cruz, Calif., after 19.6 yr of Exposure.

Top to bottom, cold drawn, Nos. 001, 004, and 005; air quenched, Nos. 002, 006, and 007. Cold-drawn wires are more corroded than air quenched. These wires are less corroded than those at Davis, Calif. (×1).

### *Findings from Tension Tests:*

Tension tests are made on the unfabricated wire specimens. These specimens are exposed in groups of seven taken from the same lot of wire and supposedly alike. An attempt is made to remove and test the first specimen of each group when it has been estimated to have lost 5 to 10 per cent of its strength, and the seventh specimen when it is estimated to have lost some 75 per cent of its strength. There were originally about 840 tensile strength specimens exposed at each site. The number of specimens removed to date is shown in Table B.

TABLE B.—NUMBER OF SPECIMENS REMOVED FOR TENSILE STRENGTH TESTS.

| Exposure Site | Number of Wires Removed 1955 and 1956 | | | | Total Removed to Date |
|---|---|---|---|---|---|
| | Bare Steel | Zinc-Coated | Lead-Coated | Other Types | |
| Pittsburgh, Pa. | 0 | 0 | 0 | 0 | 771 |
| Sandy Hook, N. J. | 0 | 0 | 0 | 0 | 462 |
| Bridgeport, Conn. | 0 | 0 | 0 | 0 | 435 |
| State College, Pa. | 0 | 0 | 0 | 0 | 185 |
| Ithaca, N. Y. | 19 | 67 | 4 | 6[a] | 156 |
| Lafayette, Ind. | 19 | 69 | 4 | 10[a] | 259 |
| Ames, Iowa | 19 | 52 | 4 | 0 | 133 |
| Manhattan, Kans. | 19 | 12 | 4 | 0 | 94 |
| College Station, Tex. | 19 | 21 | 4 | 6[a] | 107 |
| Davis, Calif. | 19 | 6 | 4 | 6[a] | 92 |
| Santa Cruz, Calif. | 19 | 14 | 4 | 6[a] | 100 |
| Total | 133 | 241 | 28 | 34 | 2794 |

[a] Corrosion-resistant steel.

TABLE C. — LOSS IN TENSILE STRENGTH, PER CENT PER YEAR OF EXPOSURE AFTER FIRST RUST.

| Exposure Site | No. 14½ Gage | No. 12½ Gage | No. 11 Gage | No. 9 Gage | No. 6 Gage |
|---|---|---|---|---|---|
| Pittsburgh, Pa. | 15 | 10 | 7 | 5 | 3 |
| Sandy Hook, N. J. | 9 | 5 | 4 | 3 | 2 |
| Bridgeport, Conn | 5 | 3 | 3 | 1 | 1 |
| State College, Pa. | 4 | 2 | 1 | 1 | ... |
| Ithaca, N. Y. | 2.2 | 1.6 | 1.1 | 0.9 | 0.6 |
| Lafayette, Ind. | 3.1 | 2.0 | 1.7 | 1.2 | 1.0 |
| Ames, Iowa | 1.1 | 0.5 | 0.4 | 0.4 | 0.6 |
| Manhattan, Kans. | 1.1 | 0.2 | 0.1 | 0.1 | 0.2 |
| College Station, Tex. | 0.8 | 0.4 | 0.5 | 0.3 | 0.2 |
| Davis, Calif. | 0.9 | 0.5 | 0.4 | 0.4 | 0.3 |
| Santa Cruz, Calif | 2.0 | 1.0 | 0.9 | 0.6 | 0.6 |

TABLE I.—REPORT OF 1955 AND 1956 INSPECTIONS OF UNFABRICATED WIRE SPECIMENS.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; MG = intermediate between M and G; Y = yellowed or rust stained but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (5, 10, 50, 100) are percentages; PHR, PHY = rusted, yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn respectively signify corroded to brown or green appearance; GY = predominantly G but showing indication of Y. All vacant blocks indicate 100R.

| Coating Group | Average Coating oz per sq ft of surface[a] | Wire | Bridgeport, Conn. | | Lafayette, Ind. | | Ithaca, N. Y. | | State College, Pa. | | Ames, Iowa | | College Station, Tex. | | Santa Cruz, Calif. | | Manhattan, Kans. | | Davis, Calif | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 18.5 yr | 19.5 yr | 19.0 yr | 20.0 yr | 18.9 yr | 19.9 yr | 18.3 yr | 19.3 yr | 18.8 yr | 19.6 yr | 18.8 yr | 19.6 yr | 18.8 yr | 19.6 yr | 18.9 yr | 19.6 yr | 18.8 yr | 19.6 yr |
| 0.20 | 0.27 | No. 512 | | | | | | | | | | | | | | | 100R | 100R | 100R | 100R |
| to | 0.25 | No. 410 | | | | | | | | | | | | | | | 100R | 100R | 100R | 100R |
| 0.30 | 0.28 | No. 411 | | | | | | | | | | | | | | | 78R | 100R | 9R | 17R |
| Avg. | 0.29 | No. 510 | | | | | | | | | | | | | | | 100R | 100R | 19R | 36R |
| 0.28 | 0.30 | No. 412 | | | | | | | | | | | | | | | 79R | 100R | 10R | 21R |
| | 0.24 | No. 511 | | | | | | | | | | | | | | | 100R | 100R | 95R | 99R |
| 0.25 | 0.25 | No. 315 | | | | | | | | | | | 100R | 100R | 100R | 100R | 100R | 100R | 100R | 100R |
| to | 0.28 | No. 215 | | | | | | | | | | | 100R | 100R | 94R | 96R | 100R | 100R | 90R | 98R |
| 0.35 | 0.27 | No. 117 | | | | | | | | | | | 100R | 100R | 89R | 94R | 100R | 100R | 45R | 68R |
| Avg. | 0.46 | No. 317 | | | | | | | | | | | 25R | 29R | GY | Y | GY | Y | G | Y |
| 0.30 | 0.29 | No. 217 | | | | | | | | | | | 100R | 100R | 80R | 94R | 100R | 100R | 3R | 3R |
| | 0.32 | No. 316 | | | | | | | | | | | 100R | 100R | 69R | 93R | 50R | 100R | 3R | 4R |
| | 0.31 | No. 216 | | | | | | | | | | | 100R | 100R | 71R | 93R | 44R | 66R | 1R | 1R |
| | 0.25 | No. 115 | | | | | | | | | | | 100R | 100R | 100R | 100R | 100R | 100R | 83R | 94R |
| 0.35 | 0.34 | No. 520 | | | | | | | | | | | 100R | 100R | 100R | 100R | 24R | 97R | 1R | 1R |
| to | 0.38 | No. 422 | | | | | | | | | | | 100R | 100R | 5R | 7R | GY | 2R | GY | Y |
| 0.45 | 0.35 | No. 423 | | | | | | | | | | | 100R | 100R | 17R | 51R | 16R | 48R | GY | Y |
| | 0.44 | No. 522 | | | | | | | | | | | 100R | 100R | 3R | 6R | GY | Y | 1R | Y |
| Avg. | 0.37 | No. 282 | | | | | | | | | | | 57R | 60R | GY | 2R | GY | 1R | GY | Y |
| 0.38 | 0.40 | No. 521 | | | | | | | | | | | 100R | 100R | 18R | 21R | 6R | 7R | 1R | Y |
| | 0.49 | No. 421 | | | | | | | | | | | 100R | 100R | 22R | 36R | 15R | 66R | GY | Y |
| 0.45 | 0.50 | No. 325 | | | | | | | | | 100R | 100R | 2R | 3R | GY | Y | GY | Y | 1R | Y |
| to | 0.49 | No. 228 | | | | | | | | | 100R | 100R | 4R | 5R | GY | Y | GY | Y | GY | Y |
| 0.55 | 0.56 | No. 329 | | | | | | | | | 100R | 100R | 11R | 11R | GY | Y | GY | Y | GY | Y |
| Avg. | 0.59 | No. 227 | | | | | | | | | 100R | 100R | 2R | 3R | GY | Y | GY | Y | GY | Y |
| 0.47 | 0.41 | No. 125 | | | | | | | | | 100R | 100R | 54R | 60R | 2R | 3R | 4R | 19R | 1R | Y |
| | 0.54 | No. 127 | | | | | | | | | 100R | 100R | 5R | 8R | GY | Y | GY | Y | 1R | Y |
| | 0.40 | No. 326 | | | | | | | | | 100R | 100R | 8R | 12R | GY | 1R | GY | 1R | G | Y |
| | 0.46 | No. 226 | | | | | | | | | 99R | 100R | 4R | 5R | GY | Y | GY | Y | GY | Y |
| | 0.48 | No. 327 | | | | | | | | | 100R | 100R | 2R | 2R | GY | Y | GY | Y | G | Y |
| | 0.45 | No. 225 | | | | | | | | | 100R | 100R | 1R | 1R | GY | Y | GY | Y | G | Y |
| 0.50 | 0.59 | No. 530 | | | | | | | | | 100R | 100R | G | G | MG | G | MG | G | 1R | 1R |
| to | 0.58 | No. 432 | | | | | | | | | 100R | 100R | G | G | MG | G | MG | G | MG | G |
| 0.60 | 0.58 | No. 531 | | | | | | | | | 100R | 100R | 1R | 2R | G | GY | GY | Y | G | Y |
| Avg. | 0.49 | No. 431 | | | | | | | | | 100R | 100R | 3R | 5R | GY | Y | GY | Y | GY | Y |
| 0.56 | 0.56 | No. 430 | | | | | | | | | 97R | 100R | 2R | 3R | GY | Y | GY | Y | G | Y |

| | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.60 | 0.67 | No. 338 | | | | | | | | | 37R | 74R | G | G | MG | G | MG | G | MG | G |
| to | 0.66 | No. 236 | | | | | | | | | 75R | 76R | G | G | MG | G | MG | G | MG | G |
| 0.70 | 0.61 | No. 335 | | | | | | | | | 100R | 100R | 2R | 4R | GY | Y | GY | Y | GY | Y |
| Avg. | 0.72 | No. 237 | | | | | | | | | 74R | 74R | GY | GY | MG | GY | MG | GY | GY | GY |
| 0.65 | 0.74 | No. 135 | | | | | | | | | 28R | 57R | GY | GY | GY | Y | GY | Y | GY | GY |
| | 0.66 | No. 337 | | | | | | | | | 100R | 100R | 7R | 14R | GY | Y | GY | Y | 1R | Y |
| | 0.82 | No. 235 | | | | | | | | | 1R | 3R | 1R | 1R | GY | Y | GY | Y | GY | Y |
| | 0.65 | No. 239 | | | | | | | | | 37R | 74R | 3R | 3R | GY | Y | GY | Y | G | Y |
| | 0.67 | No. 138 | | | | | | | | | 68R | 95R | 5R | 5R | GY | Y | GY | Y | 1R | Y |
| | 0.64 | No. 139 | | | | | | | | | 73R | 93R | 8R | 10R | GY | Y | 6R | Y | 1R | Y |
| | 0.73 | No. 336 | | | | | | | | | 94R | 100R | 3R | 4R | GY | Y | GY | Y | GY | Y |
| | 0.60 | No. 238 | | | | | | | | | 100R | 100R | 3R | 3R | GY | Y | GY | Y | GY | Y |
| 0.70 | 0.72 | No. 540 | | | | | 100R | 100R | 100R | 100R | 97R | 100R | G | G | MG | G | MG | G | MG | G |
| to | 0.65 | No. 442 | | | | | 100R | 100R | 100R | 100R | 99R | 100R | G | G | MG | G | MG | G | MG | G |
| 0.85 | 0.83 | No. 543 | | | | | 99R | 100R | 100R | 100R | 57R | 70R | GY | 1R | G | G | G | GY | G | G |
| Avg. | 0.77 | No. 441 | | | | | 100R | 100R | 100R | 100R | 77R | 96R | GY | G | G | G | MG | GY | G | G |
| 0.75 | 0.69 | No. 542 | | | | | 100R | 100R | 100R | 100R | 100R | 100R | GY | GY | MG | GY | GY | Y | G | Y |
| | 0.74 | No. 443 | | | | | 100R | 100R | 100R | 100R | 84R | 96R | G | G | MG | G | MG | GY | MG | G |
| | Cr 0.74 | No. 495 | | | | | 100R | 100R | 100R | 100R | 7R | 33R | ... | ... | ... | ... | MG | GY | ... | ... |
| | 0.80 | No. 541 | | | | | 100R | 100R | 100R | 100R | 17R | 50R | 12R | 11R | GY | Y | GY | Y | G | Y |
| | 0.72 | No. 440 | | | | | 100R | 100R | 100R | 100R | 98R | 100R | GY | 1R | GY | Y | GY | Y | GY | Y |
| | 0.87 | No. 444 | | | | | 100R | 100R | 100R | 100R | 1R | Y | 1R | 1R | GY | Y | GY | Y | G | Y |
| 0.80 | 1.04 | No. 348 | | | | | 91R | 100R | 8R | 35R | G | G | G | G | MG | G | MG | G | MG | G |
| to | 0.66 | No. 248 | | | | | 100R | 100R | 100R | 100R | 91R | 99R | G | G | MG | G | MG | G | MG | G |
| 1.00 | 0.96 | No. 347 | | | | | 100R | 100R | 100R | 100R | 7R | 16R | G | G | MG | G | MG | GY | MG | G |
| Avg. | 0.97 | No. 246 | | | | | 99R | 100R | 89R | 90R | 20R | Y | G | G | G | G | MG | GY | MG | G |
| 0.88 | 0.84 | No. 346 | | | | | 100R | 100R | 100R | 100R | 1R | Y | GY | GY | G | Y | G | Y | G | Y |
| | 0.80 | No. 245 | | | | | 100R | 100R | 100R | 100R | 11R | 27R | GY | GY | GY | Y | GY | Y | G | Y |
| | Cr 0.80 | No. 296 | | | | | 100R | 100R | 99R | 100R | G | Y | G | G | ... | ... | G | GY | ... | ... |
| | 0.90 | No. 148 | | | | | 100R | 100R | 99R | 99R | 2R | Y | GY | GY | G | Y | GY | GY | G | GY |
| | 0.85 | No. 149 | | | | | 100R | 100R | 98R | 99R | 65R | 80R | GY | GY | MG | G | MG | GY | MG | G |
| | 0.83 | No. 345 | | | | | 100R | 100R | 100R | 100R | 1R | 15R | 3R | 3R | GY | Y | GY | Y | GY | Y |
| | 0.94 | No. 247 | | | | | 100R | 100R | 99R | 100R | 1R | Y | 3R | 3R | GY | Y | GY | Y | GY | Y |
| Avg. | 1.35 | No. 800 | | | 51R | 68R | GY | 13R | GY | Y | 6R | Y | G | G | ... | ... | MG | GY | ... | ... |
| 1.28 | 1.20 | No. 801 | | | 100R | 100R | 86R | 99R | 53R | 82R | 1R | Y | G | G | ... | ... | MG | GY | ... | ... |
| 1.60 | 2.08 | No. 552 | 100R | 100R | G | G | G | G | G | G | G | G | G | G | MG | G | MG | G | MG | G |
| to | 1.71 | No. 451 | 100R | 100R | G | G | G | G | G | G | G | G | G | G | MG | G | MG | G | MG | G |
| 1.80 | 1.73 | No. 350 | 100R | 100R | GY | G | G | G | G | G | G | G | G | G | MG | G | MG | G | MG | G |
| Avg. | 1.67 | No. 252 | 100R | 100R | G | G | G | G | G | G | G | G | G | G | MG | G | MG | G | MG | G |
| 1.76 | 1.65 | No. 351 | 100R | 100R | GY | 3R | G | 3R | 1R | 1R | 1R | G | G | G | G | G | MG | G | MG | G |
| | 1.76 | No. 250 | 100R | 100R | GY | 1R | G | 1R | GY | GY | G | G | G | G | G | G | MG | G | MG | G |
| | 1.69 | No. 551 | 100R | 100R | G | GY | G | G | G | G | G | G | G | G | MG | G | MG | G | MG | G |
| | 1.48 | No. 450 | 100R | 100R | GY | 2R | G | 1R | 1R | 1R | G | G | G | G | MG | G | MG | G | MG | G |
| | 1.82 | No. 352 | 100R | 100R | GY | 2R | GY | GY | GY | GY | G | G | G | G | MG | G | MG | G | MG | G |
| | 1.68 | No. 251 | 100R | 100R | GY | 2R | GY | GY | 1R | 1R | 1R | GY | G | G | MG | G | MG | G | MG | G |
| | 1.86 | No. 151 | 90R | 100R | 25R | 32R | GY | 35R | 4R | 6R | G | GY | G | G | MG | G | MG | GY | MG | G |
| Heavy | 2.80 | No. 397 | GY | GY | G | G | G | G | G | G | G | G | G | G | MG | G | MG | G | MG | G |
| Avg. | 2.78 | No. 298 | GY | GY | G | G | G | G | G | G | G | G | G | G | MG | G | MG | G | MG | G |
| 2.89 | 3.00 | No. 299 | GY | GY | G | GY | GY | GY | G | GY | G | G | G | G | ... | ... | MG | G | ... | ... |
| Copper Covered | 7.42 | No. 021 | Gn | Gn | Gn | Gn | Gn | Gn | Gn | Gn | Br | Br | Br | Br | Br | Br | Br | Br | Br | Br |
| Wires | 9.24 | No. 022 | Gn | Gn | Gn | Gn | Gn | Gn | Gn | Gn | Br | Br | Br | Br | Br | Br | Br | Br | Br | Br |

(Continued on p. 108.)

TABLE I.—*Concluded.*

| Coating Group | Average Coating oz per sq ft of surface[a] | Wire | Bridgeport, Conn. | | Lafayette, Ind. | | Ithaca, N. Y. | | State College, Pa. | | Ames, Iowa | | College Station, Tex. | | Santa Cruz, Calif. | | Manhattan, Kans. | | Davis, Calif. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 18.5 yr | 19.5 yr | 19.0 yr | 20.0 yr | 18.9 yr | 19.9 yr | 18.3 yr | 19.3 yr | 18.8 yr | 19.6 yr | 18.8 yr | 19.6 yr | 18.8 pr | 19.6 yr | 18.9 yr | 19.6 yr | 18.8 yr | 19.6 yr |
| Lead Coated Wires | 1.40 | No. 040 | Y | Y | 32R | 41PHR | 33R | 30R | 75R | PHR | 31PHY | G,SR | 4R | 4R | 18SR | 18SR | 5PHR | GSR | 19SR | 20SR |
| | 1.48 | No. 041 | Y | Y | 14R | 19PHR | 33R | 24R | 75R | PHR | 57R | G,SR | 11R | 13R | 62SR | 52SR | 21PHR | 42R | 61SR | 29SR |
| | 1.68 | No. 042 | Y | Y | 29R | 32PHR | 23R | 21R | 37R | PHR | 47R | G,SR | 6R | 9R | 27SR | 25SR | 12PHR | 16R | 26SR | 9SR |
| | 1.90 | No. 043 | Y | Y | 13R | 27PHR | 18R | 15R | 55R | PHR | 70R | G,SR | GY | GY | 43SR | 54SR | G | GSR | 18SR | 21SR |
| 12 to 14% Cr Cold drawn | None | No. 001 | Y | Y | G | SR | D | D | G | G | SY | SR | 3R | 4R | 97SR | 9R | M | GSR | 50SR | 3R |
| | " | No. 004 | Y | Y | 1R | SR | D | D | G | G | SY | SR | 1R | 4R | 77SR | 6R | M | GSR | 44SR | 2R |
| | " | No. 005 | Y | Y | G | SR | D | D | G | G | SY | SR | 13R | 17R | 98SR | 10R | M | GSR | 57SR | 7R |
| 12 to 14% Cr Air quenched | None | No. 002 | GY | GY | G | G | D | D | G | G | SY | SR | 1R | 1R | 28SR | 28SR | M | GSR | 20SR | 60SR |
| | " | No. 006 | GY | GY | G | G | D | D | G | G | SY | SR | GY | GY | 26SR | 24SR | M | GSR | 16SR | 16SR |
| | " | No. 007 | GY | GY | G | SR | D | D | G | G | SY | SR | 4R | 4R | 67SR | 64SR | M | GSR | 41SR | 41SR |
| 18% Cr and 8% Ni | None | No. 003 | G | GY | M | M | M | M | MG | MG | SY | MG,SR | GY | MG | MG | MG | M | M | MG | MG |
| | " | No. 008 | G | G | M | M | M | M | MG | MG | SY | MG,SR | GY | MG | MG | MG | M | M | MG | MG |
| | " | No. 009 | G | GY | M | M | M | M | MG | MG | SY | MG,SR | GY | MG | MG | MG | M | M | MG | MG |
| | " | No. 015 | G | G | MG | MG | M | M | G | MG | SY | MG,SR | GY | MG | ... | ... | M | M | ... | ... |

[a] The zinc coating weights reported in this column are the stripping test determinations made on samples of unfabricated wire.

TABLE II.—REPORT OF 1955 AND 1956 INSPECTIONS OF BARBED WIRE SPECIMENS.

Abbreviations of symbols used to designate appearance or state of corrosion: M = metallic; G = gray; Y = yellowed, or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn, respectively, signify corroded to a brown or green appearance; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y; RB = rust on barbs. All vacant blocks indicate 100R.

| Coating Group | Average Coating, oz per sq ft of surface[a] | Wire[b] | State College, Pa. | | Lafayette, Ind. | | Ames, Iowa | | Manhattan, Kans. | | Ithaca, N. Y. | | Santa Cruz, Calif. | | College Station, Tex. | | Davis, Calif. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 17.4 yr | 18.4 yr | 19.0 yr | 20.0 yr | 18.8 yr | 19.6 yr | 19.0 yr | 19.6 yr | 18.9 yr | 19.9 yr | 18.8 yr | 19.8 yr | 18.8 yr | 19.9 yr | 18.8 yr | 19.8 yr |
| 0.20 to 0.30 | 0.27 | No. C410 | | | | | | | 100R | 100R | | | 100R | 100R | 100R | 100R | 60R | 85R |
| | 0.24 | No. C411 | | | | | | | 60R | 100R | | | 100R | 100R | 100R | 100R | 95R | 98R |
| | 0.27 | No. C412 | | | | | | | 40R | 65R | | | 50R | 80R | 40R | 40R | GY | 3R |
| 0.35 to 0.45 | 0.37 | No. C421 | | | | | | | GY | Y | | | 20R | 20R | 3R | 3R | GY | Y |
| | 0.44 | No. C422 | | | | | | | 20R | 10R | | | 25R | 25R | 13R | 20R | GY | 5R |
| | 0.28 | No. C420 | | | | | | | 75R | 90R | | | 10R | 60R | 35R | 43R | 3R | 8R |
| 0.50 to 0.60 | 0.46 | No. C430 | | | | | 100R | 100R | G | Y | 100R | 100R | 20R | 20R | GY | GY | GY | Y |
| | 0.53 | No. C431 | | | | | 15R | 10R | 10R,RB | Y | 85R | 100R | 25R | 25R | 15R | 20R | GY | 3R |
| | 0.53 | No. C432 | | | | | 60R | 75R | MG | G | 100R | 100R | G | G | G | GY | MG | G |
| 0.70 to 0.85 | 0.64 | No. C441 | 60R | 100R | | | 15R,RB | Y | MG | G | | | G | GY | G | GY | MG | G |
| | 0.82 | No. C442 | 95R | 100R | | | G,RB | Y | MG | G | | | G | GY | G | G | MG | GY |
| | 0.70 | No. C443 | 100R | 100R | | | 60R,RB | 15R,RB | G | GY | | | G | Y | GY | GY | G | GY |
| 0.80 to 1.00 | 0.97 | No. C445 | GY | 2R | 60R | 70R | G | Y | MG | MG | 8R | 23R | G | GY | G | G | MG | GY |
| | 0.92 | No. C446 | 10R | 50R | 50R | 75R | G,RB | Y | MG | MG | 20R | 55R | G | GY | G | G | MG | GY |
| 1.60 to 1.80 | 1.64 | No. C450 | 25R | 70R | 60R | 75R | G | Y | MG | GY | 78R | 83R | G | GY | G | G | MG | GY |
| | 1.52 | No. C452 | G | GY | 1R | 5R | G | GY | MG | G | 1R | 4R | G | G | G | G | MG | G |
| | 1.95 | No. C451 | G,RB | GY,RB | GY,RB | 5R | G,RB | Y | MG | G | 60R | 65R | G | G | G | G | MG | G |
| Heavy Coating | 1.99 | No. C494 | G | G | G | G | G | G | MG | G | GY | G | G | G | G | G | MG | G |
| | 1.99 | No. C494 | G | G | G | G | G | G | MG | G | G | G | G | G | G | G | MG | G |
| | 1.99 | No. C447 | G,RB | GY,RB | 1R,RB | 2R | G,RB | GY | MG | G | 35R | 55R | G | G | G | G | MG | G |

(Continued on p. 110.)

TABLE II.—*Concluded.*

| Coating Group | Average Coating, oz per sq ft of surface[a] | Wire[b] | State College, Pa. | | Lafayette, Ind. | | Ames, Iowa | | Manhattan, Kans. | | Ithaca, N. Y. | | Santa Cruz, Calif. | | College Station, Tex. | | Davis, Calif. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 17.4 yr | 18.4 yr | 19.0 yr | 20.0 yr | 18.8 yr | 19.6 yr | 19.0 yr | 19.6 yr | 18.9 yr | 19.9 yr | 18.8 yr | 19.8 yr | 18.8 yr | 19.9 yr | 18.8 yr | 19.8 yr |
| Copper covered (3.97 oz per sq ft) | 3.97 | No. C025 | Gn | Gn | Gn | Gn | Br | Gn | DBr | DBr | Gn,RB | Gn,RB | Gn,RB | Gn,RB | Br | Br | Br,RB | Br |
| | 3.97 | No. C025 | Gn | Gn | ... | ... | ... | ... | ... | ... | Gn,RB | Gn,RB | ... | ... | ... | ... | | ... |
| Lead-coated (1.83 oz per sq ft) | 1.83 | No. C045 | ... | ... | 10PHR | 15PHR | 30R,RB | YPHR | 10PHY | G,PHR | 40R | 20R | 33SR | 33SR | 7PHR | 5PHR | 25SR | 25SR |
| | 1.83 | No. C045 | ... | ... | 10PHR | 15PHR | 20R,RB | YPHR | ... | ... | 35R | 20R | ... | ... | 4PHR | 5PHR | ... | ... |

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of barbed wire of the specific wire lot number.

[b] These barbed wire numbers do not always correspond to the unfabricated wire numbers as seen opposite:

| Barbed Wire | Unfabricated Wire | Barbed Wire | Unfabricated Wire |
|---|---|---|---|
| No. C410 | No. 410 | No. C441 | No. 442 |
| No. C411 | ... | No. C442 | No. 441 |
| No. C412 | No. 412 | No. C443 | No. 443 |
| No. C421 | ... | No. C445 | ... |
| No. C422 | No. 421 | No. C446 | ... |
| No. C420 | No. 423 | No. C447 | ... |
| No. C430 | No. 431 | No. C450 | ... |
| No. C432 | No. 432 | No. C451 | No. 451 |
| No. C431 | No. 430 | No. C494 | ... |
| | | No. C452 | No. 450 |

## TABLE III.—REPORT OF 1955 AND 1956 INSPECTIONS OF FARM FIELD FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; Y = yellowed or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; SR, SY = speckled appearance of rust or yellowing; Br, Gn, respectively, signify corroded to a brown or green appearance; D = dark or dirt or soot excluding a better observation; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y. All vacant blocks indicate 100R.

| Coating Group | Average Coating, oz per sq ft of surface[a] | Fence | State College, Pa.[b] | | Lafayette, Ind. | | Ames, Iowa | | Manhattan, Kans. | | Ithaca, N. Y. | | Santa Cruz, Calif. | | College Station, Tex. | | Davis, Calif. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 17.4 yr | 18.4 yr | 19.0 yr | 20.0 yr | 18.8 yr | 19.6 yr | 18.9 yr | 19.6 yr | 18.9 yr | 19.9 yr | 18.8 yr | 19.8 yr | 18.8 yr | 19.9 yr | 18.9 yr | 19.8 yr |
| 0.20 | 0.27 | No. A512 | | | | | | | 100R | 100R | | | 90R | 90R | | | 95R | 100R |
| to | 0.39 | No. A410 | | | | | | | 100R | 100R | | | 75R | 80R | | | 95R | 100R |
| 0.30 | 0.28 | No. A411 | | | | | | | 50R | 90R | | | 98R | 100R | | | 63R | 73R |
| Avg. | 0.29 | No. A510 | | | | | | | 50R | 100R | | | 96R | 100R | | | 60R | 65R |
| 0.29 | 0.30 | No. A412 | | | | | | | 60R | 90R | | | 100R | 100R | | | 55R | 65R |
| | 0.23 | No. A511 | | | | | | | 50R | 95R | | | 95R | 100R | | | 100R | 100R |
| 0.25 | 0.25 | No. A315 | | | | | | | 60R | 100R | | | 95R | 100R | 100R | 100R | 100R | 100R |
| to | 0.28 | No. A215 | | | | | | | 100R | 100R | | | 93R | 95R | 100R | 100R | 100R | 100R |
| 0.35 | 0.36 | No. A317 | | | | | | | 10R | 40R | | | 40R | 50R | 43R | 45R | 8R | 10R |
| Avg. | 0.32 | No. A316 | | | | | | | 25R | 60R | | | 90R | 90R | 100R | 100R | 33R | 40R |
| 0.30 | 0.30 | No. A216 | | | | | | | 25R | 75R | | | 75R | 90R | 100R | 100R | 15R | 20R |
| 0.35 | 0.38 | No. A520 | | | | | | | 20R | 50R | | | 40R | 60R | 100R | 100R | 20R | 35R |
| to | 0.31 | No. A422 | | | | | | | 50R | 90R | | | 75R | 90R | 95R | 95R | 25R | 38R |
| 0.45 | 0.31 | No. A423 | | | | | | | 10R | 50R | | | 50R | 65R | 100R | 100R | 5R | 8R |
| Avg. | 0.44 | No. A522 | | | | | | | GY | Y | | | 35R | 35R | 45R | 50R | GY | 5R |
| 0.38 | 0.36 | No. A521 | | | | | | | 25R | 50R | | | 60R | 70R | 60R | 63R | 38R | 45R |
| | 0.49 | No. A421 | | | | | | | 10R | 10R | | | 35R | 35R | 40R | 40R | 18R | 23R |
| 0.45 | 0.42 | No. A325 | | | | | | | GY | Y | | | 25R | 25R | 8R | 8R | GY | Y |
| to | 0.50 | No. A228 | | | | | | | GY | Y | | | 30R | 35R | 10R | 10R | GY | Y |
| 0.55 | 0.51 | No. A227 | | | | | | | GY | 5R | | | 30R | 35R | 18R | 18R | GY | Y |
| Avg. | 0.35 | No. A326 | | | | | | | 20R | Y | | | 50R | 60R | 45R | 55R | GY | Y |
| 0.45 | 0.47 | No. A327 | | | | | | | GY | Y | | | 15R | 15R | 3R | 4R | GY | Y |
| | 0.46 | No. A225 | | | | | | | GY | Y | | | 18R | 18R | GY | 5R | GY | Y |

(Continued on p. 112.)

TABLE III.—*Continued.*

| Coating Group | Average Coating, oz per sq ft of surface[a] | Fence | State College, Pa.[b] | | Lafayette, Ind. | | Ames, Iowa | | Manhattan, Kans. | | Ithaca, N. Y. | | Santa Cruz, Calif. | | College Station, Tex. | | Davis, Calif. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 17.4 yr | 18.4 yr | 19.0 yr | 20.0 yr | 18.8 yr | 19.6 yr | 18.9 yr | 19.6 yr | 18.9 yr | 19.9 yr | 18.8 yr | 19.8 yr | 18.8 yr | 19.9 yr | 18.9 yr | 19.8 yr |
| 0.50 | 0.62 | No. A530 | 100R | 100R | | | 75R | 100R | MG | G | | | MG | G | G | G | MG | G |
| to | 0.60 | No. A432 | 87R | 100R | | | 55R | 60R | MG | G | | | MG | G | G | G | MG | G |
| 0.60 | 0.61 | No. A531 | 100R | 100R | | | 100R | 100R | GY | GY | | | 1SR | 8R | 6R | 6R | GY | GY |
| Avg. | 0.48 | No. A431 | 100R | 100R | | | 95R | 95R | GY | Y | | | 10R | 10R | 4R | 10R | GY | Y |
| 0.54 | 0.52 | No. A430 | 100R | 100R | | | 100R | 100R | GY | Y | | | 15R | 15R | 5R | 5R | GY | Y |
| | 0.49 | No. A532 | 100R | 100R | | | 100R | 100R | GY | Y | | | 20R | 20R | 5R | 5R | G | Y |
| | 0.49 | No. A433 | 100R | 100R | | | 100R | 100R | GY | Y | | | 15R | 15R | 10R | 10R | GY | Y |
| 0.60 | 0.69 | No. A338 | 67R | 75R | 95R | 95R | 50R | 30R | MG | G | 100R | 100R | MG | G | G | G | MG | G |
| to | 0.69 | No. A236 | 57R | 80R | 90R | 100R | G | G | MG | G | 93R | 97R | MG | G | G | G | MG | G |
| 0.70 | 0.61 | No. A335 | 100R | 100R | 100R | 100R | 95R | 30R | G | GY | 100R | 100R | 2SR | YSR | GY | GY | G | GY |
| Avg. | 0.60 | No. A237 | 98R | 98R | 100R | 100R | 90R | 60R | GY | GY | 100R | 100R | GY | YSR | 1R | 1R | MG | GY |
| 0.62 | 0.53 | No. A337 | 100R | 100R | 100R | 100R | 80R | 60R | GY | Y | 100R | 100R | 20R | 20R | 8R | 8R | GY | Y |
| | 0.63 | No. A235 | 100R | 100R | 100R | 100R | 95R | 60R | GY | Y | 100R | 100R | 18R | 18R | 10R | 10R | GY | Y |
| | 0.60 | No. A336 | 100R | 100R | 100R | 100R | 70R | 85R | GY | Y | 100R | 100R | 25R | 25R | 5R | 5R | GY | Y |
| | 0.60 | No. A238 | 100R | 100R | 100R | 100R | 10R | 60R | GY | Y | 100R | 100R | 25R | 25R | 5R | 8R | G | Y |
| 0.70 | 0.68 | No. A540 | 87R | 95R | 95R | 95R | 50R | 30R | MG | G | 100R | 100R | MG | G | G | G | MG | G |
| to | 0.86 | No. A442 | 37R | 30R | 100R | 100R | G | G | MG | G | 88R | 100R | MG | G | G | G | MG | G |
| 0.85 | 0.74 | No. A543 | 100R | 100R | 100R | 100R | 100R | 30R | MG | GY | 100R | 100R | G | G | GY | GY | MG | GY |
| Avg. | 0.76 | No. A441 | 80R | 95R | 100R | 100R | 70R | 10R | MG | GY | 100R | 100R | G | GY | G | GY | MG | G |
| 0.76 | 0.71 | No. A542 | 100R | 100R | 100R | 100R | 80R | 40R | G | GY | 100R | 100R | G | GY | GY | GY | G | GY |
| | 0.78 | No. A443 | 100R | 100R | 100R | 100R | 50R | 25R | MG | GY | 100R | 100R | MG | G | GY | GY | G | GY |
| | 0.70 | No. A541 | 95R | 100R | 100R | 100R | 70R | 55R | MG | GY | 100R | 100R | G | G | G | GY | GY | GY |
| | 0.79 | No. A440 | 92R | 98R | 100R | 100R | 75R | 50R | MG | GY | 100R | 100R | G | G | GY | GY | MG | GY |
| 0.80 | 1.06 | No. A348 | G | G | 30R | 70R | G | G | MG | G | 73R | 7R | MG | G | G | G | MG | G |
| to | 0.85 | No. A248 | GY | GY | 80R | 85R | G | G | MG | G | 13R | 45R | MG | G | G | G | MG | G |
| 1.00 | 0.86 | No. A347 | 40R | 40R | 100R | 100R | 5R | Y | MG | GY | 85R | 85R | 10R | 10R | G | GY | MG | G |
| Avg. | 0.94 | No. A246 | 63R | 65R | 100R | 100R | 15R | GY | MG | GY | 93R | 95R | G | GY | GY | GY | MG | G |
| 0.90 | 0.89 | No. A346 | 99R | 99R | 100R | 100R | 5R | YPHR | G | GY | 88R | 93R | G | 1SR | GY | GY | MG | GY |
| | 0.81 | No. A245 | 100R | 100R | 100R | 100R | G | YPHR | GY | GY | 99R | 100R | G | YSR | GY | GY | MG | GY |
| | 0.84 | No. A345 | 100R | 100R | 100R | 100R | G | YPHR | GY | Y | 94R | 100R | 10R | 10R | 3R | 3R | G | Y |
| | 0.97 | No. A247 | 83R | 85R | 100R | 100R | G | YPHR | GY | Y | 95R | 97R | 8R | 8R | 8R | 8R | GY | Y |

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1.60 to 1.80 | 1.76 | No. A350 | G | G | G | G | G | G | MG | G | G | G | MG | G | G | G | MG | G |
|  | 1.71 | No. A252 | G | G | G | G | G | G | MG | G | G | G | MG | G | G | G | MG | G |
|  | 1.68 | No. A352 | 1R | 1R | 3R | 3R | G | GY | MG | G | G | GY | G | G | G | GY | MG | G |
| Avg. 2.75 | 2.79 | No. A397 | G | G | G | G | G | G | MG | G | G | G | MG | G | G | G | MG | G |
|  | 2.71 | No. A298 | G | G | G | G | G | G | MG | G | G | G | MG | G | G | G | MG | G |
| 12–14% Cr, cold drawn |  | No. A001 | G | G | MG | G | SY | GSR | MG | GPHR | G | D | 10R | 10R | 2R | 8R | 40SR | 40R |
| 12–14% Cr, air quenched |  | No. A002 | MG | G | MG | MG | SY | G | M | GPHR | G | D | 5R | 5R | 2R | 4R | 80SR | 38R |
| 18% Cr, 8% Ni |  | No. A003 | MG | MG | M | M | M | M | M | M | M | M | MG | MG | MG | MG | MG | MG |
| Copper covered (7.42 oz per sq ft) |  | No. A020 | Gn | Gn | Gn | Gn | Br | Gn | Br | Br | Gn | Gn | Gn | Gn | Br | Br | Br | Br |
| Lead coated (1.48 oz per sq ft) |  | No. A040 | 100PHR | PHR | 10PHR | 25PHR | 30R | YPHR | 10R | 25R | 10PHR | 25PHR | 49SR | 50SR | 15R | 18PHR | 40SR | 40SR |

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[a] The zinc coating weights reported in this column are the stripping test determinations made on samples of fence of the specific wire lot number.

[b] The rating for the state of corrosion is based on the appearance of the top half of the fence. At all locations the bottom part of the fence shows less corrosion than the top part.

TABLE IV.—REPORT OF 1955 AND 1956 INSPECTIONS OF CHAIN LINK FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; Y = yellowed or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; Br, Gn, respectively, signify corroded to a brown or green appearance; SR, SY = speckled appearance of rust or yellow; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y. All vacant blocks indicate 100R.

All 200 series wires are 9 gage. Lead-coated wire, 9 gage. Corrosion-resistant steel wire, 14 gage.

All 100 series wires are 6 gage. Copper-covered wire, 0.128 in. diameter.

| Zinc Coating, oz per sq ft of surface, by Stripping Test | Fence | Bridgeport, Conn.[c] | | State College, Pa. | | Lafayette, Ind. | | Ithaca, N. Y. | | Santa Cruz, Calif. | | Davis, Calif. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 18.5 yr | 19.5 yr | 17.4 yr | 18.4 yr | 19.0 yr | 20 yr | 18.9 yr | 19.9yr | 18.8 yr | 19.8 yr | 18.9 yr | 19.9 yr |
| 0.29 | No. E280[a] | * | | | | | | | | 100R | 100R | 65R | 75R |
| 0.52 | No. E281 | * | | | | | | | | 10R | 10R | 5SR | Y |
| 0.37 | No. E282 | * | | | | | | | | 20R | 30R | 6R | 15R |
| 1.22 | No. E185 | * | | Y | 20R | 80R | 90R | 13R | 78R | MG | G | MG | G |
| 1.13 | No. E285 | * | | 80R | 90R | 100R | 100R | 88R | 93R | MG | G | MG | GY |
| 2.92 | No. E290 | GY | GY | G | G | G | G | G | G | MG | G | MG | G |
| 2.09 | No. E291 | 5R | 10R | 2R | 2R | G | G | G | G | MG | G | MG | G |
| 1.60 | No. H280[a] | * | | 33R | 55R | 60R | 75R | 75R | 73R | G | G | MG | G |
| 1.83 | No. H185 | * | | GY | GY | 70R | 90R | 28R | 75R | MG | G | MG | GY |
| 1.60 | No. H186 | * | | GY | GY | 60R | 75R | GY | 65R | G | G | MG | G |
| 1.61 | No. H187 | * | | GY | GY | 30R | 35R | GY | 8R | G | G | MG | G |
| 1.55 | No. H188 | * | | 33R | 40R | 90R | 95R | 60R | 85R | MG | G | MG | G |
| 1.36 | No. H285 | * | | 10R | 30R | 50R | 75R | 80R | 90R | G | G | MG | G |
| 1.97 | No. H286 | * | | GY | 10R | 40R | 50R | 50R | 97R | MG | GY | MG | GY |
| 1.46 | No. H287 | * | | 10R | 20R | 50R | 70R | 53R | 70R | MG | G | MG | G |
| 1.74 | No. H288 | * | | GY | Y | 10R | 25R | GY | 4R | G | G | MG | GY |
| 1.40 | No. H289[b] | * | | G | G | G | G | G | G | MG | G | MG | G |
| 4.21 | No. H290 | Y | 10R | GY | GY | GY | GY | GY | 8R | MG | MG | MG | G |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lead-coated (1.75 oz per sq ft) | No. L050 | Y | Y | 40PHR | PHR | 50R | 50PHR | 13R | 23R | 35SR | 35SR | 35SR | 20SR |
| Coppered-covered (8.52 oz per sq ft) | No. K030 | Gn | Gn | Gn | Gn | Gn | Gn | Gn | Gn | Br | Br | Br | Br |
| Corrosion-resistant steel (18% Cr–8% Ni) | No. J011 | MG | MG | M | M | MG | MG | M | M | MG | MG | MG | MG |

[a] E numbers, coating applied before weaving; H numbers, coating applied after weaving.

[b] Although this sample has an H number, its appearance indicates that it was galvanized before weaving.

[c] The rating for the state of corrosion is based on the appearance of the fence at eye level.

* Specimens destroyed shortly after the 16.5 yr inspection, all 100R.

TABLE V.—REPORT OF 1955 AND 1956 INSPECTIONS OF WIRE STRAND SPECIMENS.

Abbreviations and symbols used to designate appearance or state of corrosion: M = metallic; G = gray; Y = yellowed or rust-stained, but not showing actual rough rust of base metal; R = rough rust of base metal, figures preceding R (as 5, 20, 50, 100) are percentages; PHR, PHY = rusted or yellowed in pinholes; Br, Gn, respectively, signify corroded to a brown or green appearance; SR, SY = speckled appearance of R or Y; MG = intermediate between M and G; GY = predominantly gray but showing indication of Y.

| Zinc Coating, oz per sq ft of surface | Strand | Bridgeport, Conn. | | State College, Pa. | | Lafayette, Ind. | | Ames, Iowa | | Manhattan, Kans. | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 18.5 yr | 19.5 yr | 18.3 yr | 19.3 yr | 19.0 yr | 20.0 yr | 18.8 yr | 19.6 yr | 18.9 yr | 19.6 yr |
| 0.69 | No. D345 | 100R | 100R | GY | 3R | 15R | 25R | G | G | MG | G |
| 0.88 | No. D346 | 100R | 100R | GY | 1R | 3R | 8R | G | GY | MG | G |
| 0.87 | No. D347 | 100R | 100R | GY | 5R | 40R | 33R | G | GY | MG | GY |
| 0.93 | No. D348 | 100R | 100R | GY | 1R | 20R | 25R | G | G | MG | G |
| 1.10 | No. D360 | 100R | 100R | G | G | 2R | 5R | G | G | MG | G |
| 1.17 | No. D361 | 100R | 100R | GY | GY | 3R | 8R | G | GY | MG | GY |
| 1.03 | No. D362 | 5R | 95R | GY | 1R | 18R | 35R | G | Y | MG | GY |
| 1.51 | No. D365 | 100R | 100R | GY | GY | 6R | 10R | G | GY | MG | GY |
| 1.39 | No. D366 | 25R | 30R | G | G | G | G | G | G | MG | G |
| 1.32 | No. D367 | 100R | 100R | Y | Y | 1R | 5R | G | Y | MG | GY |
| 1.71 | No. D371 | GY | GY | G | G | G | G | G | G | MG | G |
| 2.47 | No. D375 | GY | GY | G | G | G | G | G | G | MG | G |
| Lead-coated (1.96 oz per sq ft) | No. D045 | Y | Y | 100PHR | PHR | 40PHR | 50PHR | 68R | YPHR | 8R | 23R |

| Zinc Coating, oz per sq ft of surface | Strand | Ithaca, N. Y. | | Santa Cruz, Calif. | | College Station, Tex. | | Davis, Calif. | |
|---|---|---|---|---|---|---|---|---|---|
| | | 18.9 yr | 19.9 yr | 18.8 yr | 19.8 yr | 18.8 yr | 19.9 yr | 18.9 yr | 19.8 yr |
| 0.69 | No. D345 | 35R | 55R | G | GY | G | GY | G | GY |
| 0.88 | No. D346 | 2R | 2R | MG | MG | G | G | MG | G |
| 0.87 | No. D347 | 5R | 30R | G | GY | G | G | G | G |
| 0.83 | No. D348 | 15R | 11R | MG | G | G | G | MG | G |
| 1.10 | No. D360 | G | G | MG | G | G | G | MG | G |
| 1.17 | No. D361 | Y | 5R | G | GY | G | G | G | G |
| 1.03 | No. D362 | Y | 4R | MG | G | G | G | MG | G |
| 1.51 | No. D365 | GY | 3R | G | GY | G | G | G | G |
| 1.39 | No. D366 | G | G | MG | G | G | G | MG | G |
| 1.32 | No. D367 | 3R | 8R | G | G | G | G | MG | G |
| 1.71 | No. D371 | G | G | MG | G | G | G | MG | G |
| 2.47 | No. D375 | G | G | G | G | G | G | MG | G |
| Lead-coated (1.96 oz per sq ft) | No. D045 | 20R | 28R | 68SR | 68SR | 10PHR | 20PHR | 45SR | 10R |

TABLE VI.—LOSS OF TENSILE STRENGTH OF WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT AMES, IOWA.

500 Series (14½ gage); 400 Series (12½ gage); 300 Series (11 Gage); 200 Series (9 Gage); 100 Series (6 Gage) Unfabricated Wires.

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 500*[a] | 500 | 0 | 2.1 | 9.0 | 4.3 | 3.0 | 10.0 | 3.3 | 11.0 | 26.0 | 2.4 | 19.6 | 33.0 | 1.7 |
| No. 506* | 520 | 0 | 2.1 | 11.5 | 5.5 | 3.0 | 11.5 | 3.8 | 11.0 | 30.8 | 2.8 | 19.6 | 36.5 | 1.9 |
| No. 507* | 590 | 0 | 2.1 | 8.4 | 4.0 | 3.0 | 9.3 | 3.1 | 11.0 | 31.0 | 2.8 | 19.6 | 27.7 | 1.4 |
| No. 510 | 500 | 7.4 | 12.2 | 13.0 | 1.1 | | | | | | | | | |
| No. 511 | 280 | 6.2 | 13.4 | 19.6 | 1.5 | | | | | | | | | |
| No. 512 | 320 | 5.5 | 14.1 | 21.9 | 1.6 | | | | | | | | | |
| No. 520 | 280 | 7.3 | 12.3 | 1.8 | 0.1 | | | | | | | | | |
| No. 521 | 270 | 8.9 | 10.7 | 3.7 | 0.3 | | | | | | | | | |
| No. 522 | 500 | 9.6 | 10.0 | 14.0 | 1.4 | | | | | | | | | |
| No. 530 | 310 | 13.1 | 6.5 | 12.9 | 2.0 | | | | | | | | | |
| No. 531 | 500 | 12.0 | 7.6 | 12.0 | 1.6 | | | | | | | | | |
| No. 540 | 300 | 15.9 | 3.7 | 1.7 | 0.5 | | | | | | | | | |
| No. 542 | 510 | 14.9 | 4.7 | 3.9 | 0.8 | | | | | | | | | |
| No. 543 | 300 | 13.0 | 6.6 | −1.7 | ... | | | | | | | | | |
| No. 400*[a] | 470 | 0 | 2.1 | 4.2 | 2.0 | 4.1 | 6.3 | 1.5 | 11.0 | 14.6 | 1.3 | 19.6 | 15.6 | 0.8 |
| No. 401*[a] | 440 | 0 | 2.1 | 1.1 | 0.5 | 4.1 | 7.7 | 1.9 | 11.0 | 18.9 | 1.7 | 19.6 | 16.7 | 0.9 |
| No. 405* | 470 | 0 | 2.1 | 4.1 | 2.0 | 4.1 | 9.2 | 2.2 | 11.0 | 21.7 | 2.0 | 19.6 | 21.7 | 1.1 |
| No. 406* | 460 | 0 | 2.1 | 4.2 | 2.0 | 4.1 | 5.3 | 1.3 | 11.0 | 13.8 | 1.3 | 19.6 | 13.8 | 0.7 |
| No. 410 | 470 | 5.5 | 14.1 | −6.4 | ... | | | | | | | | | |
| No. 411 | 630 | 6.7 | 12.9 | 8.7 | 0.7 | | | | | | | | | |
| No. 412 | 840 | 6.7 | 12.9 | 13.1 | 1.0 | | | | | | | | | |
| No. 421 | 470 | 7.9 | 11.7 | 2.1 | 0.2 | | | | | | | | | |
| No. 422 | 500 | 8.8 | 10.8 | 4.0 | 0.4 | | | | | | | | | |
| No. 423 | 400 | 7.7 | 11.9 | 7.5 | 0.6 | | | | | | | | | |
| No. 430 | 780 | 11.9 | 7.7 | −3.2 | ... | | | | | | | | | |
| No. 431 | 880 | 11.7 | 7.9 | 10.2 | 1.3 | | | | | | | | | |
| No. 432 | 510 | 14.0 | 5.6 | 4.9 | 0.9 | | | | | | | | | |
| No. 441 | 520 | 12.7 | 6.9 | −8.7 | ... | | | | | | | | | |
| No. 442 | 470 | 16.7 | 2.9 | 2.1 | 0.7 | | | | | | | | | |
| No. 443 | 750 | 13.7 | 5.9 | −2.7 | ... | | | | | | | | | |
| No 444 | 570 | 19.3 | 0.3 | −0.9 | ... | | | | | | | | | |

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. 300*[a] | 730 | 0 | 2.1 | 4.0 | 1.9 | 4.1 | 6.7 | 1.6 | 11.0 | 10.8 | 1.0 | 19.6 | 5.4 | 0.3 |
| No. 302*[a] | 1110 | 0 | 2.1 | 9.6 | 4.6 | 4.1 | 11.4 | 2.8 | 11.0 | 16.8 | 1.5 | 19.6 | 24.5 | 1.3 |
| No. 305* | 1070 | 0 | 2.1 | 4.6 | 2.2 | 4.1 | 6.0 | 1.5 | 11.0 | 15.2 | 1.4 | 19.6 | 18.4 | 0.9 |
| No. 306* | 700 | 0 | 2.1 | 4.2 | 2.0 | 4.1 | 5.7 | 1.4 | 11.0 | 10.5 | 1.0 | 19.6 | 13.4 | 0.7 |
| No. 315 | 890 | 5.3 | 14.3 | 7.9 | 0.6 | | | | | | | | | |
| No. 316 | 1110 | 7.7 | 11.9 | 4.1 | 0.3 | | | | | | | | | |
| No. 317 | 780 | 9.6 | 10.0 | 3.8 | 0.4 | | | | | | | | | |
| No. 325 | 730 | 11.0 | 8.6 | −6.8 | ... | | | | | | | | | |
| No. 326 | 700 | 9.9 | 9.7 | 5.0 | 0.5 | | | | | | | | | |
| No. 327 | 1160 | 11.9 | 7.7 | −3.0 | ... | | | | | | | | | |
| No. 329 | 720 | 9.9 | 9.7 | 4.9 | 0.5 | | | | | | | | | |
| No. 336 | 760 | 12.0 | 7.6 | 2.0 | 0.3 | | | | | | | | | |
| No. 337 | 860 | 11.3 | 8.3 | −3.4 | ... | | | | | | | | | |
| No. 200*[a] | 1010 | 0 | 2.1 | 7.7 | 3.7 | 4.1 | 8.0 | 2.0 | 11.0 | 10.4 | 0.9 | 19.6 | 15.8 | 0.8 |
| No. 202*[a] | 1640 | 0 | 2.1 | 3.3 | 1.6 | 4.1 | 4.3 | 1.0 | 19.6 | 15.5 | 0.8 | | | |
| No. 205* | 1110 | 0 | 2.1 | 1.8 | 0.9 | 4.1 | 1.0 | 0.2 | 11.0 | 5.9 | 0.5 | 19.6 | 10.4 | 0.5 |
| No. 206* | 1480 | 0 | 2.1 | 6.1 | 2.9 | 4.1 | 7.4 | 1.8 | 11.0 | 12.5 | 1.1 | 19.6 | 12.8 | 0.7 |
| No. 215 | 1200 | 5.5 | 14.1 | 6.7 | 0.5 | | | | | | | | | |
| No. 216 | 1630 | 7.7 | 11.9 | 6.1 | 0.5 | | | | | | | | | |
| No. 217 | 1080 | 7.7 | 11.9 | −1.4 | ... | | | | | | | | | |
| No. 225 | 1650 | 12.9 | 6.7 | 0.3 | 0.05 | | | | | | | | | |
| No. 226 | 1150 | 11.0 | 8.6 | −0.4 | ... | | | | | | | | | |
| No. 227 | 1640 | 11.0 | 8.6 | 5.8 | 0.7 | | | | | | | | | |
| No. 228 | 1080 | 9.9 | 9.7 | 1.9 | 0.2 | | | | | | | | | |
| No. 236 | 1270 | 16.7 | 2.9 | −20.9 | ... | | | | | | | | | |
| No. 237 | 1540 | 13.3 | 6.3 | 1.3 | 0.2 | | | | | | | | | |
| No. 238 | 1650 | 12.7 | 6.9 | 0.9 | 0.1 | | | | | | | | | |
| No. 248 | 1450 | 16.6 | 3.0 | 2.1 | 0.7 | | | | | | | | | |
| No. 101*[a] | 2330 | 0 | 2.1 | 4.5 | 2.1 | 4.1 | 3.8 | 0.9 | 11.0 | 7.1 | 0.6 | 19.6 | 3.0 | 0.2 |
| No. 105*[a] | 2750 | 0 | 2.1 | 4.2 | 2.0 | 4.1 | 2.7 | 0.7 | 11.0 | 9.4 | 0.9 | 19.6 | 6.9 | 0.4 |
| No. 102* | 2110 | 0 | 2.1 | 4.5 | 2.1 | 4.1 | 8.5 | 2.1 | 11.0 | 9.9 | 0.9 | 19.6 | 10.0 | 0.5 |
| No. 106* | 2430 | 0 | 2.1 | −0.6 | ... | 4.1 | −0.8 | ... | 11.0 | 7.1 | 0.6 | 19.6 | 6.0 | 0.3 |
| No. 115 | 2690 | 5.3 | 14.3 | 3.2 | 0.2 | | | | | | | | | |
| No. 117 | 2230 | 6.5 | 13.1 | 49.8 | 3.8 | | | | | | | | | |
| No. 125 | 1840 | 8.8 | 10.8 | 3.5 | 0.3 | | | | | | | | | |
| No. 127 | 2540 | 11.0 | 8.6 | 3.9 | 0.5 | | | | | | | | | |
| No. 138 | 1960 | 11.0 | 8.6 | −2.6 | ... | | | | | | | | | |
| No. 139 | 1920 | 11.0 | 8.6 | −6.0 | ... | | | | | | | | | |
| No. 149 | 1910 | 14.1 | 5.5 | −1.0 | ... | | | | | | | | | |

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* Uncoated wires. All other wires were zinc coated.

[a] Low copper content wires, 0.05 to 0.08 per cent Cu. All other wires have approximately 0.25 per cent Cu.

[b] Summarizes data thru 1956.

TABLE VII.—LOSS OF TENSILE STRENGTH OF WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT MANHATTAN, KANS.
500 Series (14½ gage); 400 Series (12½ gage); 300 Series (11 gage); 200 Series (9 gage); 100 Series (6 gage) Unfabricated Wires.

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 501*[a] | 500 | 0 | 1.9 | 1.0 | 0.5 | 3.9 | 5.0 | 1.3 | 10.9 | 7.0 | 0.6 | 19.6 | −8.0 | ... |
| No. 506* | 520 | 0 | 1.9 | 1.0 | 0.5 | 3.9 | 5.8 | 1.5 | 10.9 | 5.8 | 0.5 | 19.6 | 3.0 | 0.2 |
| No. 507* | 590 | 0 | 1.9 | −4.2 | ... | 3.9 | 0.8 | 0.2 | 10.9 | 3.4 | 0.3 | 19.6 | 21.0 | 1.1 |
| No. 510 | 500 | 10.7 | 8.9 | 46.0 | 5.2 | | | | | | | | | |
| No. 511 | 280 | 10.7 | 8.9 | −5.4 | ... | | | | | | | | | |
| No. 512 | 320 | 10.5 | 9.1 | 3.1 | 0.3 | | | | | | | | | |
| No. 400*[a] | 470 | 0 | 1.9 | −1.0 | ... | 3.9 | −2.0 | ... | 10.9 | 1.0 | 0.1 | 19.6 | 3.2 | 0.2 |
| No. 401*[a] | 440 | 0 | 1.9 | −3.3 | ... | 3.9 | −4.4 | ... | 10.9 | −1.0 | ... | 19.6 | 4.5 | 0.2 |
| No. 405* | 470 | 0 | 1.9 | −10.6 | ... | 3.9 | −5.0 | ... | 10.9 | −3.0 | ... | 19.6 | 4.3 | 0.2 |
| No. 406* | 460 | 0 | 1.9 | −3.0 | ... | 3.9 | −4.0 | ... | 10.9 | −2.0 | ... | 19.6 | 3.3 | 0.2 |
| No. 410 | 470 | 9.9 | 9.7 | −23.4 | ... | | | | | | | | | |
| No. 411 | 630 | 10.8 | 8.8 | 3.2 | 0.4 | | | | | | | | | |
| No. 412 | 840 | 13.9 | 5.7 | 1.8 | 0.3 | | | | | | | | | |
| No. 300*[a] | 730 | 0 | 1.9 | −2.0 | ... | 3.9 | −5.0 | ... | 10.9 | −3.0 | ... | 19.6 | 1.4 | 0.1 |
| No. 302*[a] | 1110 | 0 | 1.9 | 3.2 | 1.7 | 3.9 | 4.1 | 1.1 | 10.9 | 5.9 | 0.5 | 19.6 | 8.0 | 0.4 |
| No. 305* | 1070 | 0 | 1.9 | −1.5 | ... | 3.9 | −1.0 | ... | 10.9 | 0.0 | 0.0 | 19.6 | 2.3 | 0.1 |
| No. 306* | 700 | 0 | 1.9 | −3.0 | ... | 3.9 | −4.0 | ... | 10.9 | −1.4 | ... | 19.6 | 1.4 | 0.1 |
| No. 315 | 890 | 8.9 | 10.7 | −1.7 | ... | | | | | | | | | |
| No. 316 | 1110 | 13.9 | 5.7 | −0.5 | ... | | | | | | | | | |
| No. 200*[a] | 1010 | 0 | 1.9 | 0.0 | 0.0 | 3.9 | 0.0 | 0.0 | 10.9 | −1.0 | ... | 19.6 | 2.5 | 0.1 |
| No. 202*[a] | 1640 | 0 | 1.9 | −0.3 | ... | 3.9 | 0.6 | 0.2 | 10.9 | ... | ... | 19.6 | 1.5 | 0.1 |
| No. 205* | 1110 | 0 | 1.9 | 0.0 | 0.0 | 3.9 | 1.8 | 0.5 | 10.9 | 1.0 | 0.1 | 19.6 | 5.0 | 0.3 |
| No. 206* | 1480 | 0 | 1.9 | 1.0 | 0.5 | 3.9 | 3.0 | 0.8 | 10.9 | 3.4 | 0.3 | 19.6 | 3.0 | 0.2 |
| No. 215 | 1200 | 9.5 | 10.1 | 0.4 | 0.04 | | | | | | | | | |
| No. 217 | 1080 | 11.1 | 8.5 | −11.6 | ... | | | | | | | | | |
| No. 101*[a] | 2330 | 0 | 1.9 | 1.4 | 0.7 | 3.9 | 1.0 | 0.3 | 10.9 | 1.5 | 0.1 | 19.6 | 3.6 | 0.2 |
| No. 105*[a] | 2750 | 0 | 1.9 | 5.3 | 2.8 | 3.9 | 5.0 | 1.3 | 10.9 | 0.0 | 0.0 | 19.6 | 4.0 | 0.2 |
| No. 102* | 2110 | 0 | 1.9 | 3.5 | 1.8 | 3.9 | 6.6 | 1.7 | 10.9 | 6.6 | 0.6 | 19.6 | 6.9 | 0.4 |
| No. 106* | 2430 | 0 | 1.9 | −0.6 | ... | 3.9 | −3.0 | ... | 10.9 | 1.4 | 0.1 | 19.6 | 0.4 | 0.02 |
| No. 115 | 2690 | 8.9 | 10.7 | 2.2 | 0.2 | | | | | | | | | |
| No. 117 | 2230 | 8.9 | 10.7 | 2.2 | 0.2 | | | | | | | | | |

* Uncoated wires. All other wires were zinc coated.

[a] Low copper content wire, 0.05 to 0.08 per cent Cu. All other wires have approximately 0.25 per cent Cu.

[b] Summarizes data through 1956.

TABLE VIII.—LOSS OF TENSILE STRENGTH OF WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT COLLEGE STATION, TEX.
500 Series (14½ Gage); 400 Series (12½ Gage); 300 Series (11 Gage); 200 Series (9 Gage); 100 Series (6 Gage) Unfabricated Wires.

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 501*[a] | 500 | 0 | 1.9 | 8.0 | 4.2 | 3.8 | 6.0 | 1.6 | 10.8 | 24.0 | 2.2 | 19.6 | 32.0 | 1.6 |
| No. 506* | 520 | 0 | 1.9 | 3.0 | 1.6 | 3.8 | 8.6 | 2.3 | 10.8 | 24.0 | 2.2 | 19.6 | 35.6 | 1.8 |
| No. 507* | 590 | 0 | 1.9 | 3.4 | 1.8 | 3.8 | 9.2 | 2.4 | 10.8 | 26.1 | 2.4 | 19.6 | 37.3 | 1.9 |
| No. 510 | 500 | 10.6 | 9.0 | 4.0 | 0.4 | | | | | | | | | |
| No. 511 | 280 | 6.9 | 12.7 | 3.6 | 0.3 | | | | | | | | | |
| No. 512 | 320 | 6.3 | 13.3 | 14.1 | 1.1 | | | | | | | | | |
| No. 520 | 280 | 11.6 | 8.0 | −10.7 | ... | | | | | | | | | |
| No. 521 | 270 | 9.8 | 9.8 | −7.4 | ... | | | | | | | | | |
| No. 522 | 500 | 10.0 | 9.6 | −13.0 | ... | | | | | | | | | |
| No. 400*[a] | 470 | 0 | 1.9 | 0.0 | 0.0 | 3.8 | 2.1 | 0.6 | 10.8 | 9.4 | 0.9 | 19.6 | 17.0 | 0.9 |
| No. 401*[a] | 440 | 0 | 1.9 | −2.0 | ... | 3.8 | 3.3 | 0.9 | 10.8 | 14.4 | 1.3 | 19.6 | 19.3 | 1.0 |
| No. 405* | 470 | 0 | 1.9 | 0.0 | 0.0 | 3.8 | 2.1 | 0.6 | 10.8 | 13.0 | 1.2 | 19.6 | 16.0 | 0.8 |
| No. 406* | 460 | 0 | 1.9 | 1.0 | 0.5 | 3.8 | 3.2 | 0.8 | 10.8 | 8.5 | 0.8 | 19.6 | 13.0 | 0.7 |
| No. 410 | 470 | 6.0 | 13.6 | −13.8 | ... | | | | | | | | | |
| No. 411 | 630 | 10.8 | 8.8 | −2.4 | ... | | | | | | | | | |
| No. 412 | 840 | 10.8 | 8.8 | 3.6 | 0.4 | | | | | | | | | |
| No. 421 | 470 | 10.9 | 8.7 | 0.0 | 0.0 | | | | | | | | | |
| No. 422 | 500 | 11.0 | 8.6 | −4.0 | ... | | | | | | | | | |
| No. 423 | 400 | 10.9 | 8.7 | −3.8 | ... | | | | | | | | | |
| No. 300*[a] | 730 | 0 | 1.9 | 0.7 | 0.4 | 3.8 | 2.0 | 0.5 | 10.8 | 7.4 | 0.7 | 19.6 | 13.0 | 0.7 |
| No. 302*[a] | 1110 | 0 | 1.9 | 4.5 | 2.4 | 3.8 | 8.6 | 2.3 | 10.8 | 19.1 | 1.8 | 19.6 | 18.5 | 0.9 |
| No. 305* | 1070 | 0 | 1.9 | 2.0 | 1.0 | 3.8 | 5.5 | 1.5 | 10.8 | 12.0 | 1.1 | 19.6 | 14.5 | 0.7 |
| No. 306* | 700 | 0 | 1.9 | −0.7 | ... | 3.8 | −2.1 | ... | 10.8 | 7.1 | 0.7 | 19.6 | 11.8 | 0.6 |
| No. 315 | 890 | 6.5 | 13.1 | 3.9 | 0.3 | | | | | | | | | |
| No. 316 | 1110 | 10.8 | 8.8 | −0.9 | ... | | | | | | | | | |

(Continued on p. 122.)

TABLE VII.—LOSS OF TENSILE STRENGTH OF WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT MANHATTAN, KANS.
500 Series (14½ gage); 400 Series (12½ gage); 300 Series (11 gage); 200 Series (9 gage); 100 Series (6 gage) Unfabricated Wires.

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 501*[a] | 500 | 0 | 1.9 | 1.0 | 0.5 | 3.9 | 5.0 | 1.3 | 10.9 | 7.0 | 0.6 | 19.6 | −8.0 | ... |
| No. 506* | 520 | 0 | 1.9 | 1.0 | 0.5 | 3.9 | 5.8 | 1.5 | 10.9 | 5.8 | 0.5 | 19.6 | 3.0 | 0.2 |
| No. 507* | 590 | 0 | 1.9 | −4.2 | ... | 3.9 | 0.8 | 0.2 | 10.9 | 3.4 | 0.3 | 19.6 | 21.0 | 1.1 |
| No. 510 | 500 | 10.7 | 8.9 | 46.0 | 5.2 | | | | | | | | | |
| No. 511 | 280 | 10.7 | 8.9 | −5.4 | ... | | | | | | | | | |
| No. 512 | 320 | 10.5 | 9.1 | 3.1 | 0.3 | | | | | | | | | |
| No. 400*[a] | 470 | 0 | 1.9 | −1.0 | ... | 3.9 | −2.0 | ... | 10.9 | 1.0 | 0.1 | 19.6 | 3.2 | 0.2 |
| No. 401*[a] | 440 | 0 | 1.9 | −3.3 | ... | 3.9 | −4.4 | ... | 10.9 | −1.0 | ... | 19.6 | 4.5 | 0.2 |
| No. 405* | 470 | 0 | 1.9 | −10.6 | ... | 3.9 | −5.0 | ... | 10.9 | −3.0 | ... | 19.6 | 4.3 | 0.2 |
| No. 406* | 460 | 0 | 1.9 | −3.0 | ... | 3.9 | −4.0 | ... | 10.9 | −2.0 | ... | 19.6 | 3.3 | 0.2 |
| No. 410 | 470 | 9.9 | 9.7 | −23.4 | ... | | | | | | | | | |
| No. 411 | 630 | 10.8 | 8.8 | 3.2 | 0.4 | | | | | | | | | |
| No. 412 | 840 | 13.9 | 5.7 | 1.8 | 0.3 | | | | | | | | | |
| No. 300*[a] | 730 | 0 | 1.9 | −2.0 | ... | 3.9 | −5.0 | ... | 10.9 | −3.0 | ... | 19.6 | 1.4 | 0.1 |
| No. 302*[a] | 1110 | 0 | 1.9 | 3.2 | 1.7 | 3.9 | 4.1 | 1.1 | 10.9 | 5.9 | 0.5 | 19.6 | 8.0 | 0.4 |
| No. 305* | 1070 | 0 | 1.9 | −1.5 | ... | 3.9 | −1.0 | ... | 10.9 | 0.0 | 0.0 | 19.6 | 2.3 | 0.1 |
| No. 306* | 700 | 0 | 1.9 | −3.0 | ... | 3.9 | −4.0 | ... | 10.9 | −1.4 | ... | 19.6 | 1.4 | 0.1 |
| No. 315 | 890 | 8.9 | 10.7 | −1.7 | ... | | | | | | | | | |
| No. 316 | 1110 | 13.9 | 5.7 | −0.5 | ... | | | | | | | | | |
| No. 200*[a] | 1010 | 0 | 1.9 | 0.0 | 0.0 | 3.9 | 0.0 | 0.0 | 10.9 | −1.0 | ... | 19.6 | 2.5 | 0.1 |
| No. 202*[a] | 1640 | 0 | 1.9 | −0.3 | ... | 3.9 | 0.6 | 0.2 | 10.9 | ... | ... | 19.6 | 1.5 | 0.1 |
| No. 205* | 1110 | 0 | 1.9 | 0.0 | 0.0 | 3.9 | 1.8 | 0.5 | 10.9 | 1.0 | 0.1 | 19.6 | 5.0 | 0.3 |
| No. 206* | 1480 | 0 | 1.9 | 1.0 | 0.5 | 3.9 | 3.0 | 0.8 | 10.9 | 3.4 | 0.3 | 19.6 | 3.0 | 0.2 |
| No. 215 | 1200 | 9.5 | 10.1 | 0.4 | 0.04 | | | | | | | | | |
| No. 217 | 1080 | 11.1 | 8.5 | −11.6 | ... | | | | | | | | | |
| No. 101*[a] | 2330 | 0 | 1.9 | 1.4 | 0.7 | 3.9 | 1.0 | 0.3 | 10.9 | 1.5 | 0.1 | 19.6 | 3.6 | 0.2 |
| No. 105*[a] | 2750 | 0 | 1.9 | 5.3 | 2.8 | 3.9 | 5.0 | 1.3 | 10.9 | 0.0 | 0.0 | 19.6 | 4.0 | 0.2 |
| No. 102* | 2110 | 0 | 1.9 | 3.5 | 1.8 | 3.9 | 6.6 | 1.7 | 10.9 | 6.6 | 0.6 | 19.6 | 6.9 | 0.4 |
| No. 106* | 2430 | 0 | 1.9 | −0.6 | ... | 3.9 | −3.0 | ... | 10.9 | 1.4 | 0.1 | 19.6 | 0.4 | 0.02 |
| No. 115 | 2690 | 8.9 | 10.7 | 2.2 | 0.2 | | | | | | | | | |
| No. 117 | 2230 | 8.9 | 10.7 | 2.2 | 0.2 | | | | | | | | | |

* Uncoated wires. All other wires were zinc coated.

[a] Low copper content wire, 0.05 to 0.08 per cent Cu. All other wires have approximately 0.25 per cent Cu.

[b] Summarizes data through 1956.

TABLE VIII.—LOSS OF TENSILE STRENGTH OF WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT COLLEGE STATION, TEX.

500 Series (14½ Gage); 400 Series (12½ Gage); 300 Series (11 Gage); 200 Series (9 Gage); 100 Series (6 Gage) Unfabricated Wires.

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 501*[a] | 500 | 0 | 1.9 | 8.0 | 4.2 | 3.8 | 6.0 | 1.6 | 10.8 | 24.0 | 2.2 | 19.6 | 32.0 | 1.6 |
| No. 506* | 520 | 0 | 1.9 | 3.0 | 1.6 | 3.8 | 8.6 | 2.3 | 10.8 | 24.0 | 2.2 | 19.6 | 35.6 | 1.8 |
| No. 507* | 590 | 0 | 1.9 | 3.4 | 1.8 | 3.8 | 9.2 | 2.4 | 10.8 | 26.1 | 2.4 | 19.6 | 37.3 | 1.9 |
| No. 510 | 500 | 10.6 | 9.0 | 4.0 | 0.4 | | | | | | | | | |
| No. 511 | 280 | 6.9 | 12.7 | 3.6 | 0.3 | | | | | | | | | |
| No. 512 | 320 | 6.3 | 13.3 | 14.1 | 1.1 | | | | | | | | | |
| No. 520 | 280 | 11.6 | 8.0 | −10.7 | ... | | | | | | | | | |
| No. 521 | 270 | 9.8 | 9.8 | −7.4 | ... | | | | | | | | | |
| No. 522 | 500 | 10.0 | 9.6 | −13.0 | ... | | | | | | | | | |
| No. 400*[a] | 470 | 0 | 1.9 | 0.0 | 0.0 | 3.8 | 2.1 | 0.6 | 10.8 | 9.4 | 0.9 | 19.6 | 17.0 | 0.9 |
| No. 401*[a] | 440 | 0 | 1.9 | −2.0 | ... | 3.8 | 3.3 | 0.9 | 10.8 | 14.4 | 1.3 | 19.6 | 19.3 | 1.0 |
| No. 405* | 470 | 0 | 1.9 | 0.0 | 0.0 | 3.8 | 2.1 | 0.6 | 10.8 | 13.0 | 1.2 | 19.6 | 16.0 | 0.8 |
| No. 406* | 460 | 0 | 1.9 | 1.0 | 0.5 | 3.8 | 3.2 | 0.8 | 10.8 | 8.5 | 0.8 | 19.6 | 13.0 | 0.7 |
| No. 410 | 470 | 6.0 | 13.6 | −13.8 | ... | | | | | | | | | |
| No. 411 | 630 | 10.8 | 8.8 | −2.4 | ... | | | | | | | | | |
| No. 412 | 840 | 10.8 | 8.8 | 3.6 | 0.4 | | | | | | | | | |
| No. 421 | 470 | 10.9 | 8.7 | 0.0 | 0.0 | | | | | | | | | |
| No. 422 | 500 | 11.0 | 8.6 | −4.0 | ... | | | | | | | | | |
| No. 423 | 400 | 10.9 | 8.7 | −3.8 | ... | | | | | | | | | |
| No. 300*[a] | 730 | 0 | 1.9 | 0.7 | 0.4 | 3.8 | 2.0 | 0.5 | 10.8 | 7.4 | 0.7 | 19.6 | 13.0 | 0.7 |
| No. 302*[a] | 1110 | 0 | 1.9 | 4.5 | 2.4 | 3.8 | 8.6 | 2.3 | 10.8 | 19.1 | 1.8 | 19.6 | 18.5 | 0.9 |
| No. 305* | 1070 | 0 | 1.9 | 2.0 | 1.0 | 3.8 | 5.5 | 1.5 | 10.8 | 12.0 | 1.1 | 19.6 | 14.5 | 0.7 |
| No. 306* | 700 | 0 | 1.9 | −0.7 | ... | 3.8 | −2.1 | ... | 10.8 | 7.1 | 0.7 | 19.6 | 11.8 | 0.6 |
| No. 315 | 890 | 6.5 | 13.1 | 3.9 | 0.3 | | | | | | | | | |
| No. 316 | 1110 | 10.8 | 8.8 | −0.9 | ... | | | | | | | | | |

(Continued on p. 122.)

[a] Low copper content wire, 0.05 to 0.08 per cent Cu. All other wires have approximately 0.25 per cent Cu.

[b] Summarizes data through 1956.

TABLE VIII.—*Concluded*

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test |  |  | Second Test |  |  | Third Test |  |  | Fourth Test |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  |  | Exposure Since 1R, yr | Tensile Strength Loss |  | Exposure Since 1R, yr | Tensile Strength Loss |  | Exposure Since 1R, yr | Tensile Strength Loss |  | Exposure Since 1R, yr | Tensile Strength Loss |  |
|  |  |  |  | Per cent | Per cent per year |  | Per cent | Per cent per year |  | Per cent | Per cent per year |  | Per cent | Per cent per year |
| No. 200*[a] | 1010 | 0 | 1.9 | −1.0 | ... | 3.8 | −1.5 | ... | 10.8 | 8.4 | 0.8 | 19.6 | 11.4 | 0.6 |
| No. 202*[a] | 1640 | 0 | 1.9 | 3.7 | 2.0 | 3.8 | 3.0 | 0.8 | 10.8 | 6.1 | 0.6 | 19.6 | 10.1 | 0.5 |
| No. 205* | 1110 | 0 | 1.9 | 0.2 | 0.1 | 3.8 | 2.7 | 0.7 | 10.8 | 3.2 | 0.3 | 19.6 | 11.3 | 0.6 |
| No. 206* | 1480 | 0 | 1.9 | 2.4 | 1.3 | 3.8 | 2.4 | 0.6 | 10.8 | 8.5 | 0.8 | 19.6 | 10.8 | 0.6 |
| No. 215 | 1200 | 7.3 | 12.3 | 1.3 | 0.1 |  |  |  |  |  |  |  |  |  |
| No. 216 | 1630 | 11.6 | 8.0 | 1.9 | 0.2 |  |  |  |  |  |  |  |  |  |
| No. 217 | 1080 | 9.3 | 10.3 | −0.5 | ... |  |  |  |  |  |  |  |  |  |
| No. 282 | 1320 | 9.2 | 10.4 | −4.9 | ... |  |  |  |  |  |  |  |  |  |
| No. 101*[a] | 2330 | 0 | 1.9 | 1.5 | 0.8 | 3.8 | 4.9 | 1.3 | 10.8 | 3.9 | 0.4 | 19.6 | 6.6 | 0.3 |
| No. 105*[a] | 2750 | 0 | 1.9 | 2.0 | 1.0 | 3.8 | 5.1 | 1.3 | 10.8 | 1.6 | 0.1 | 19.6 | 7.5 | 0.4 |
| No. 102* | 2110 | 0 | 1.9 | 6.4 | 3.4 | 3.8 | 7.6 | 2.0 | 10.8 | 9.2 | 0.9 | 19.6 | 9.2 | 0.5 |
| No. 106* | 2430 | 0 | 1.9 | −0.6 | ... | 3.8 | 2.7 | 0.7 | 10.8 | 8.4 | 0.8 | 19.6 | 6.0 | 0.3 |
| No. 115 | 2690 | 7.5 | 12.1 | −0.2 | ... |  |  |  |  |  |  |  |  |  |
| No. 117 | 2230 | 7.0 | 12.6 | 1.6 | 0.1 |  |  |  |  |  |  |  |  |  |
| No. 125 | 1840 | 12.0 | 7.6 | −1.6 | ... |  |  |  |  |  |  |  |  |  |

* Uncoated wires. All other wires were zinc coated.

[a] Low copper content wire, 0.05 to 0.08 per cent Cu. All other wires have approximately 0.25 per cent Cu.

[b] Summarizes data through 1956.

TABLE IX.—LOSS OF TENSILE STRENGTH OF WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT DAVIS, CALIF.

500 Series (14½ Gage); 400 Series (12½ Gage); 300 Series (11 Gage); 200 Series (9 Gage); 100 Series (6 Gage) Unfabricated Wires.

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 501*[a] | 500 | 0 | 1.9 | 1.0 | 0.5 | 3.8 | 1.0 | 0.3 | 10.9 | 15.0 | 1.4 | 19.6 | 24.0 | 1.2 |
| No. 506* | 520 | 0 | 1.9 | 3.0 | 1.1 | 3.8 | 2.8 | 0.7 | 10.9 | 19.2 | 1.8 | 19.6 | 24.0 | 1.2 |
| No. 507* | 590 | 0 | 1.9 | −0.8 | ... | 3.8 | 0.0 | 0.0 | 10.9 | 18.5 | 1.7 | 19.6 | 25.4 | 1.3 |
| No. 511 | 280 | 12.4 | 7.2 | 0.0 | 0.0 | | | | | | | | | |
| No. 512 | 320 | 11.7 | 7.9 | 6.7 | 0.8 | | | | | | | | | |
| No. 400*[a] | 470 | 0 | 1.9 | −2.4 | ... | 3.8 | 0.0 | 0.0 | 10.9 | 2.1 | 0.2 | 19.6 | 11.7 | 0.6 |
| No. 401*[a] | 440 | 0 | 1.9 | −5.7 | ... | 3.8 | −4.5 | ... | 10.9 | 5.7 | 0.5 | 19.6 | 11.4 | 0.6 |
| No. 405* | 470 | 0 | 1.9 | −1.0 | ... | 3.8 | −1.0 | ... | 10.9 | 7.4 | 0.7 | 19.6 | 14.0 | 0.7 |
| No. 406* | 460 | 0 | 1.9 | −1.0 | ... | 3.8 | −4.3 | ... | 10.9 | 5.4 | 0.5 | 19.6 | 14.1 | 0.7 |
| No. 410 | 470 | 11.3 | 8.3 | −21.3 | ... | | | | | | | | | |
| No. 300*[a] | 730 | 0 | 1.9 | −0.7 | ... | 3.8 | −1.4 | ... | 10.9 | 3.4 | 0.3 | 19.6 | 9.6 | 0.5 |
| No. 302*[a] | 1110 | 0 | 1.9 | 5.4 | 2.8 | 3.8 | 8.1 | 2.1 | 10.9 | 14.0 | 1.3 | 19.6 | 8.1 | 0.4 |
| No. 305* | 1070 | 0 | 1.9 | −0.5 | ... | 3.8 | −0.5 | ... | 10.9 | 6.1 | 0.6 | 19.6 | 9.8 | 0.5 |
| No. 306* | 700 | 0 | 1.9 | −1.4 | ... | 3.8 | −2.8 | ... | 10.9 | 3.6 | 0.3 | 19.6 | 8.6 | 0.4 |
| No. 315 | 890 | 11.8 | 7.8 | −0.6 | ... | | | | | | | | | |
| No. 200*[a] | 1010 | 0 | 1.9 | −1.5 | ... | 3.8 | −1.5 | ... | 10.9 | 6.0 | 0.6 | 19.6 | 10.4 | 0.5 |
| No. 202*[a] | 1640 | 0 | 1.9 | 0.3 | 0.2 | 3.8 | 3.7 | 1.0 | 10.9 | 9.5 | 0.9 | 19.6 | 11.9 | 0.6 |
| No. 205* | 1110 | 0 | 1.9 | 1.0 | 0.5 | 3.8 | −1.0 | ... | 10.9 | 6.3 | 0.6 | 19.6 | 7.7 | 0.4 |
| No. 206* | 1480 | 0 | 1.9 | 1.3 | 0.9 | 3.8 | 2.3 | 0.6 | 10.9 | 8.8 | 0.8 | 19.6 | 10.5 | 0.5 |
| No. 215 | 1200 | 12.8 | 6.8 | −0.4 | ... | | | | | | | | | |
| No. 101*[a] | 2330 | 0 | 1.9 | 1.5 | 0.8 | 3.8 | −0.5 | ... | 10.9 | 6.0 | 0.6 | 19.6 | 5.8 | 0.3 |
| No. 105*[a] | 2750 | 0 | 1.9 | −0.5 | ... | 3.8 | 1.8 | 0.5 | 10.9 | 2.4 | 0.2 | 19.6 | 4.7 | 0.2 |
| No. 102* | 2110 | 0 | 1.9 | 5.7 | 3.0 | 3.8 | 3.8 | 1.0 | 10.9 | 9.7 | 0.9 | 19.6 | 9.2 | 0.5 |
| No. 106* | 2430 | 0 | 1.9 | 0.6 | 0.3 | 3.8 | −0.8 | ... | 10.9 | 4.9 | 0.4 | 19.6 | 5.8 | 0.3 |
| No. 115 | 2690 | 12.1 | 7.5 | −0.2 | ... | | | | | | | | | |

* Uncoated wires. All other wires are zinc coated.

[a] Low copper content wire, 0.05 to 0.08 per cent Cu. All other wires have approximately 0.25 per cent Cu.

[b] Summarizes data through 1956.

TABLE X.—LOSS OF TENSILE STRENGTH OF WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT SANTA CRUZ, CALIF.

500 Series ($14\frac{1}{2}$ Gage); 400 Series ($12\frac{1}{2}$ Gage); 300 Series (11 Gage); 200 Series (9 Gage); 100 Series (6 Gage) Unfabricated Wires.

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | | Exposure Since 1R, yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 501*[a] | 500 | 0 | 1.9 | 5.0 | 2.6 | 3.8 | 7.0 | 1.8 | 10.9 | 35.0 | 3.2 | 19.6 | 52.0 | 2.7 |
| No. 506* | 520 | 0 | 1.9 | 4.8 | 2.5 | 3.8 | 8.7 | 2.3 | 10.9 | 32.7 | 3.0 | 19.6 | 51.9 | 2.7 |
| No. 507* | 590 | 0 | 1.9 | −0.8 | . . . | 3.8 | 10.2 | 2.7 | 10.9 | 40.4 | 3.7 | 19.6 | 59.3 | 3.0 |
| No. 510 | 500 | 12.8 | 6.8 | 9.0 | 1.3 | | | | | | | | | |
| No. 511 | 280 | 12.8 | 6.8 | 10.7 | 1.6 | | | | | | | | | |
| No. 512 | 320 | 12.5 | 7.1 | 15.6 | 2.2 | | | | | | | | | |
| No. 520 | 280 | 12.8 | 6.8 | 1.8 | 0.3 | | | | | | | | | |
| No. 400*[a] | 470 | 0 | 1.9 | −1.1 | . . . | 3.8 | −1.1 | . . . | 10.9 | 18.1 | 1.7 | 19.6 | 36.2 | 1.8 |
| No. 401*[a] | 440 | 0 | 1.9 | −5.7 | . . . | 3.8 | 1.1 | 0.3 | 10.9 | 15.9 | 1.5 | 19.6 | 35.2 | 1.8 |
| No. 405* | 470 | 0 | 1.9 | 0.0 | 0.0 | 3.8 | −2.1 | . . . | 10.9 | 15.9 | 1.5 | 19.6 | 31.9 | 1.6 |
| No. 406* | 460 | 0 | 1.9 | 0.0 | 0.0 | 3.8 | −1.1 | . . . | 10.9 | 13.1 | 1.2 | 19.6 | 31.5 | 1.6 |
| No. 410 | 470 | 12.8 | 6.8 | −13.8 | . . . | | | | | | | | | |
| No. 411 | 630 | 12.8 | 6.8 | −4.0 | . . . | | | | | | | | | |
| No. 412 | 840 | 12.8 | 6.8 | 0.6 | 0.1 | | | | | | | | | |
| No. 300*[a] | 730 | 0 | 1.9 | 1.4 | 0.7 | 3.8 | 14.4 | 3.8 | 10.9 | 8.9 | 0.8 | 19.6 | 22.6 | 1.2 |
| No. 302*[a] | 1110 | 0 | 1.9 | 5.9 | 3.1 | 3.8 | 8.1 | 2.1 | 10.9 | 18.5 | 1.7 | 19.6 | 31.1 | 1.6 |
| No. 305* | 1070 | 0 | 1.9 | 0.5 | 0.3 | 3.8 | 2.3 | 0.6 | 10.9 | 12.6 | 2.1 | 19.6 | 21.5 | 1.1 |
| No. 306* | 700 | 0 | 1.9 | −0.7 | . . . | 3.8 | −0.7 | . . . | 10.9 | 12.1 | 1.1 | 19.6 | 14.3 | 0.7 |
| No. 315 | 890 | 11.0 | 8.6 | 4.5 | 0.5 | | | | | | | | | |
| No. 316 | 1110 | 12.8 | 6.8 | −1.4 | . . . | | | | | | | | | |
| No. 200*[a] | 1010 | 0 | 1.9 | 0.9 | 0.5 | 3.8 | 0.0 | 0.0 | 10.9 | 10.9 | 1.0 | 19.6 | 18.8 | 1.0 |
| No. 202*[a] | 1640 | 0 | 1.9 | −2.1 | . . . | 3.8 | −4.0 | . . . | 10.9 | 7.9 | 0.7 | 19.6 | 17.4 | 0.9 |
| No. 205* | 1110 | 0 | 1.9 | 2.2 | 1.2 | 3.8 | 1.7 | 0.4 | 10.9 | 9.5 | 0.9 | 19.6 | 15.3 | 0.8 |
| No. 206* | 1480 | 0 | 1.9 | 4.0 | 2.1 | 3.8 | 1.0 | 0.3 | 10.9 | 10.8 | 1.0 | 19.6 | 18.6 | 1.0 |
| No. 215 | 1200 | 11.8 | 7.8 | 1.7 | 0.2 | | | | | | | | | |
| No. 216 | 1630 | 13.4 | 6.2 | 1.9 | 0.3 | | | | | | | | | |
| No. 217 | 1080 | 12.8 | 6.8 | −4.2 | . . . | | | | | | | | | |

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. 101*[a] | 2330 | 0 | 1.9 | −0.6 | ... | 3.8 | 0.0 | 0.0 | 10.9 | 5.4 | 0.5 | 19.6 | 13.7 | 0.7 |
| No. 105*[a] | 2750 | 0 | 1.9 | 3.8 | 2.0 | 3.8 | 3.3 | 0.9 | 10.9 | 7.8 | 0.7 | 19.6 | 10.9 | 0.6 |
| No. 102* | 2110 | 0 | 1.9 | 6.6 | 3.5 | 3.8 | 5.7 | 1.5 | 10.9 | 11.4 | 1.0 | 19.6 | 16.6 | 0.8 |
| No. 106* | 2430 | 0 | 1.9 | 3.5 | 1.8 | 3.8 | 3.9 | 1.0 | 10.9 | 6.6 | 0.6 | 19.6 | 8.9 | 0.5 |
| No. 115 | 2690 | 11.8 | 7.8 | 3.7 | 0.5 | | | | | | | | | |
| No. 117 | 2230 | 12.8 | 6.8 | 4.0 | 0.6 | | | | | | | | | |

* Uncoated wires. All other wires were zinc coated.

[a] Low copper content wire, 0.05 to 0.08 per cent Cu. All other wires have approximately 0.25 per cent Cu.

[b] Summarizes data through 1956.

TABLE XI.—LOSS OF TENSILE STRENGTH OF WIRES AFTER VARIOUS PERIODS OF EXPOSURE[b] AT LAFAYETTE, IND.
500 Series (14½ Gage); 400 Series (12½ Gage); 300 Series (11 Gage); 200 Series (9 Gage); 100 Series (6 Gage) Unfabricated Wires.

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | | Fifth Test | | | Sixth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 501*[a] | 500 | 0 | 2.0 | 13.0 | 6.5 | 3.1 | 22.0 | 7.0 | 4.1 | 25.0 | 6.1 | 10.9 | 40.0 | 3.7 | 17.8 | 54.0 | 3.0 | 20.0 | 58.0 | 2.9 |
| No. 506* | 520 | 0 | 2.0 | 11.5 | 5.8 | 3.1 | 21.2 | 6.8 | 4.1 | 23.1 | 5.6 | 10.9 | 42.3 | 3.9 | 17.8 | 60.6 | 3.4 | 20.0 | 61.5 | 3.1 |
| No. 507* | 590 | 0 | 2.0 | 14.3 | 7.2 | 3.1 | 21.0 | 6.8 | 4.1 | 22.6 | 5.5 | 10.9 | 42.0 | 3.8 | 17.8 | 62.2 | 3.5 | 20.0 | 64.4 | 3.2 |
| No. 510 | 500 | 5.2 | 12.6 | 42.0 | 3.3 | 14.8 | 49.0 | 3.3 | | | | | | | | | | | | |
| No. 511 | 280 | 4.6 | 13.2 | 46.4 | 3.5 | 15.4 | 51.8 | 3.4 | | | | | | | | | | | | |
| No. 512 | 320 | 3.9 | 13.9 | 43.8 | 3.2 | 16.1 | 57.8 | 3.6 | | | | | | | | | | | | |
| No. 520 | 280 | 5.2 | 12.6 | 30.4 | 2.4 | 14.8 | 33.9 | 2.3 | | | | | | | | | | | | |
| No. 521 | 270 | 5.5 | 12.3 | 31.5 | 2.6 | 14.5 | 42.6 | 2.9 | | | | | | | | | | | | |
| No. 522 | 500 | 6.2 | 11.6 | 39.0 | 3.4 | 13.8 | 43.0 | 3.1 | | | | | | | | | | | | |
| No. 530 | 310 | 9.9 | 7.9 | 38.7 | 4.9 | 10.1 | 37.1 | 3.7 | | | | | | | | | | | | |
| No. 531 | 500 | 7.8 | 10.0 | 38.0 | 3.8 | 12.2 | 39.0 | 3.2 | | | | | | | | | | | | |
| No 540 | 300 | 11.5 | 6.3 | 28.3 | 4.5 | 8.5 | 33.3 | 3.9 | | | | | | | | | | | | |
| No. 541 | 350 | 8.5 | 9.3 | 34.3 | 3.7 | 11.5 | 37.1 | 3.2 | | | | | | | | | | | | |
| No. 542 | 510 | 8.5 | 9.3 | 33.3 | 3.6 | 11.5 | 37.3 | 3.2 | | | | | | | | | | | | |
| No. 543 | 300 | 9.0 | 8.8 | 25.0 | 2.8 | 11.0 | 23.3 | 2.1 | | | | | | | | | | | | |
| No. 400*[a] | 470 | 0 | 2.0 | 8.4 | 4.2 | 3.1 | 10.4 | 3.4 | 4.1 | 10.4 | 2.5 | 10.9 | 21.9 | 2.0 | 17.8 | 30.2 | 1.7 | 20.0 | 33.0 | 1.7 |
| No. 401*[a] | 440 | 0 | 2.0 | 10.0 | 5.0 | 3.1 | 11.1 | 3.6 | 4.1 | 12.2 | 3.0 | 10.9 | 28.9 | 2.7 | 17.8 | 43.2 | 2.4 | 20.0 | 42.0 | 2.1 |
| No. 405* | 470 | 0 | 2.0 | 10.4 | 5.2 | 3.1 | 12.4 | 4.0 | 4.1 | 15.5 | 3.8 | 10.9 | 25.8 | 2.4 | 17.8 | 41.2 | 2.3 | 20.0 | 41.5 | 2.1 |
| No. 406* | 460 | 0 | 2.0 | 7.4 | 3.7 | 3.1 | 8.5 | 2.7 | 4.1 | 9.6 | 2.3 | 10.9 | 23.4 | 2.1 | 17.8 | 33.0 | 1.9 | 20.0 | 35.9 | 1.8 |
| No. 410 | 470 | 4.0 | 13.8 | 14.0 | 1.0 | 16.0 | 18.1 | 1.1 | | | | | | | | | | | | |
| No. 411 | 630 | 5.0 | 12.8 | 25.4 | 2.0 | 15.0 | 27.0 | 1.8 | | | | | | | | | | | | |
| No. 412 | 840 | 5.0 | 12.8 | 29.8 | 2.3 | 15.0 | 31.5 | 2.1 | | | | | | | | | | | | |
| No. 421 | 470 | 5.3 | 12.5 | 27.7 | 2.2 | 14.7 | 31.9 | 2.2 | | | | | | | | | | | | |
| No. 422 | 500 | 5.9 | 11.9 | 19.0 | 1.6 | 14.1 | 25.0 | 1.8 | | | | | | | | | | | | |
| No. 423 | 400 | 5.3 | 12.5 | 30.0 | 2.4 | 14.2 | 31.3 | 2.2 | | | | | | | | | | | | |
| No. 430 | 780 | 8.3 | 9.5 | 20.5 | 2.2 | 11.7 | 21.8 | 1.9 | | | | | | | | | | | | |
| No. 431 | 880 | 6.7 | 11.1 | 26.1 | 2.4 | 13.3 | 28.4 | 2.1 | | | | | | | | | | | | |
| No. 432 | 510 | 10.0 | 7.8 | 15.7 | 2.0 | 10.0 | 21.6 | 2.2 | | | | | | | | | | | | |
| No. 440 | 590 | 8.5 | 9.3 | 22.0 | 2.4 | 11.5 | 23.7 | 2.1 | | | | | | | | | | | | |
| No. 441 | 520 | 10.6 | 7.2 | 14.4 | 2.0 | 9.4 | 16.3 | 1.7 | | | | | | | | | | | | |
| No. 442 | 470 | 12.0 | 5.8 | 20.2 | 3.5 | 8.0 | 26.6 | 3.3 | | | | | | | | | | | | |
| No. 443 | 750 | 9.6 | 8.2 | 20.0 | 2.4 | 10.4 | 18.7 | 1.8 | | | | | | | | | | | | |
| No. 444 | 570 | 9.5 | 8.3 | 7.9 | 1.0 | 10.5 | 20.2 | 1.9 | | | | | | | | | | | | |
| No. 495 | 750 | 10.8 | 7.0 | 16.7 | 2.4 | 9.2 | 20.7 | 2.3 | | | | | | | | | | | | |

| | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. 300*[a] | 730 | 0 | 2.0 | 6.8 | 3.4 | 3.1 | 7.4 | 2.4 | 4.1 | 7.5 | 1.8 | 10.9 | 15.5 | 1.4 | 17.8 | 22.3 | 1.3 | 20.0 | 21.2 | 1.1 |
| No. 302*[a] | 1110 | 0 | 2.0 | 12.3 | 6.2 | 3.1 | 13.6 | 4.4 | 4.1 | 16.4 | 4.0 | 10.9 | 25.0 | 2.3 | 17.8 | 31.0 | 1.7 | 20.0 | 35.1 | 1.8 |
| No. 305* | 1070 | 0 | 2.0 | 7.0 | 3.5 | 3.1 | 10.1 | 3.3 | 4.1 | 10.6 | 2.6 | 10.9 | 22.1 | 2.0 | 17.8 | 27.7 | 1.6 | 20.0 | 29.9 | 1.5 |
| No. 306* | 700 | 0 | 2.0 | 5.6 | 2.8 | 3.1 | 7.8 | 2.5 | 4.1 | 7.7 | 1.9 | 10.9 | 15.5 | 1.4 | 17.8 | 21.1 | 1.2 | 20.0 | 22.9 | 1.1 |
| No. 315 | 890 | 3.8 | 14.0 | 19.1 | 1.4 | 16.2 | 23.6 | 1.5 | | | | | | | | | | | | |
| No. 316 | 1110 | 5.1 | 12.7 | 19.4 | 1.5 | 14.9 | 23.4 | 1.6 | | | | | | | | | | | | |
| No. 317 | 780 | 6.4 | 11.4 | 13.5 | 1.2 | 13.6 | 18.6 | 1.4 | | | | | | | | | | | | |
| No. 325 | 730 | 7.0 | 10.8 | 11.0 | 1.0 | 13.0 | 13.7 | 1.1 | | | | | | | | | | | | |
| No. 326 | 700 | 6.5 | 11.3 | 20.7 | 1.8 | 13.5 | 24.3 | 1.8 | | | | | | | | | | | | |
| No. 327 | 1160 | 7.6 | 10.2 | 22.8 | 2.2 | 12.4 | 20.3 | 1.6 | | | | | | | | | | | | |
| No. 329 | 720 | 6.0 | 11.8 | 31.2 | 2.6 | 14.0 | 33.3 | 2.4 | | | | | | | | | | | | |
| No. 335 | 1040 | 7.7 | 10.1 | 12.0 | 1.2 | 12.3 | 19.7 | 1.6 | | | | | | | | | | | | |
| No. 336 | 760 | 8.0 | 9.8 | 21.1 | 2.1 | 12.0 | 30.3 | 2.5 | | | | | | | | | | | | |
| No. 337 | 860 | 8.0 | 9.8 | 18.6 | 1.9 | 12.0 | 19.2 | 1.6 | | | | | | | | | | | | |
| No. 338 | 1020 | 10.8 | 7.0 | 11.7 | 1.7 | 9.2 | 14.7 | 1.6 | | | | | | | | | | | | |
| No. 345 | 890 | 9.0 | 8.8 | 23.1 | 2.6 | 11.0 | 21.4 | 1.9 | | | | | | | | | | | | |
| No. 346 | 1070 | 10.9 | 6.9 | 7.9 | 1.1 | 9.1 | 12.6 | 1.4 | | | | | | | | | | | | |
| No. 347 | 860 | 12.8 | 5.0 | 11.1 | 2.2 | 7.2 | 11.0 | 1.5 | | | | | | | | | | | | |
| No. 348 | 980 | 17.8 | 2.2 | 7.1 | 3.2 | .... | .... | ... | | | | | | | | | | | | |
| No. 200*[a] | 1010 | 0 | 2.0 | 9.1 | 4.6 | 3.1 | 10.9 | 3.5 | 10.9 | 19.3 | 1.8 | 17.8 | 18.7 | 1.1 | 20.0 | 23.3 | 1.2 | | | |
| No. 202*[a] | 1640 | 0 | 2.0 | 8.2 | 4.1 | 3.1 | 5.2 | 1.7 | 10.9 | .... | ... | 17.8 | 23.8 | 1.3 | 20.0 | 25.9 | 1.3 | | | |
| No. 205* | 1110 | 0 | 2.0 | 7.7 | 3.9 | 3.1 | 5.9 | 1.9 | 10.9 | 14.0 | 1.3 | 17.8 | 24.8 | 1.4 | 20.0 | 23.0 | 1.2 | | | |
| No. 206* | 1480 | 0 | 2.0 | 7.1 | 3.6 | 3.1 | 9.5 | 3.1 | 10.9 | 17.9 | 1.6 | 17.8 | 25.7 | 1.4 | 20.0 | 22.6 | 1.1 | | | |
| No. 215 | 1200 | 4.0 | 13.8 | 19.6 | 1.4 | 16.0 | 13.8 | 0.9 | | | | | | | | | | | | |
| No. 216 | 1630 | 5.2 | 12.6 | 21.2 | 1.7 | 14.8 | 15.6 | 1.1 | | | | | | | | | | | | |
| No. 217 | 1080 | 5.0 | 12.8 | 15.3 | 1.2 | 15.0 | 11.6 | 0.8 | | | | | | | | | | | | |
| No. 282 | 1320 | 5.0 | 12.8 | 11.7 | 0.9 | 15.0 | 14.8 | 1.0 | | | | | | | | | | | | |
| No. 225 | 1650 | 7.9 | 9.9 | 18.2 | 1.8 | 12.1 | 13.0 | 1.1 | | | | | | | | | | | | |
| No. 226 | 1150 | 7.9 | 9.9 | 18.7 | 1.9 | 12.1 | 15.2 | 1.3 | | | | | | | | | | | | |
| No. 227 | 1640 | 7.9 | 9.9 | 17.7 | 1.8 | 12.1 | 14.0 | 1.2 | | | | | | | | | | | | |
| No. 228 | 1080 | 6.0 | 11.8 | 15.3 | 1.3 | 14.0 | 11.1 | 0.8 | | | | | | | | | | | | |
| No. 235 | 1680 | 9.5 | 8.3 | 13.7 | 1.7 | 10.5 | 30.7 | 2.9 | | | | | | | | | | | | |
| No. 236 | 1270 | 10.0 | 7.8 | 1.6 | 0.2 | 10.0 | 10.2 | 1.0 | | | | | | | | | | | | |
| No. 237 | 1540 | 8.2 | 9.6 | 10.1 | 1.1 | 11.8 | 7.2 | 0.6 | | | | | | | | | | | | |
| No. 238 | 1650 | 8.0 | 9.8 | 14.3 | 1.5 | 12.0 | 13.6 | 1.1 | | | | | | | | | | | | |
| No. 239 | 1120 | 8.7 | 9.1 | 13.8 | 1.5 | 11.3 | 10.3 | 0.9 | | | | | | | | | | | | |
| No. 245 | 1570 | 9.9 | 7.9 | 11.8 | 1.5 | 10.1 | 10.2 | 1.0 | | | | | | | | | | | | |
| No. 246 | 1190 | 10.9 | 9.1 | 4.6 | 0.5 | ... | ... | ... | | | | | | | | | | | | |
| No. 247 | 1790 | 9.9 | 7.9 | 20.9 | 2.6 | 10.1 | 17.9 | 1.8 | | | | | | | | | | | | |
| No. 248 | 1450 | 11.5 | 6.3 | 6.9 | 1.1 | 8.5 | 10.7 | 1.3 | | | | | | | | | | | | |
| No. 296 | 1570 | 12.0 | 5.8 | 8.6 | 1.5 | 8.0 | 9.9 | 1.2 | | | | | | | | | | | | |

**(Continued on p. 128.)**

TABLE XI.—*Concluded.*

| Wire Lot | Original Breaking Load, lb | Exposure Until First Rust, yr | First Test | | | Second Test | | | Third Test | | | Fourth Test | | | Fifth Test | | | Sixth Test | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | | Exposure Since, 1R yr | Tensile Strength Loss | |
| | | | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year | | Per cent | Per cent per year |
| No. 101*[a] | 2330 | 0 | 2.0 | 5.7 | 2.9 | 3.1 | 5.2 | 1.7 | 10.9 | 10.6 | 1.0 | 17.8 | 18.6 | 1.0 | 20.0 | 17.8 | 0.9 | | | |
| No. 105*[a] | 2750 | 0 | 2.0 | 7.2 | 3.6 | 3.1 | 8.0 | 2.6 | 10.9 | 12.7 | 1.2 | 17.8 | 19.0 | 1.1 | 20.0 | 20.5 | 1.0 | | | |
| No. 102* | 2110 | 0 | 2.0 | 10.7 | 5.4 | 3.1 | 8.6 | 2.8 | 10.9 | 13.8 | 1.3 | 17.8 | 19.6 | 1.1 | 20.0 | 22.3 | 1.1 | | | |
| No. 106* | 2430 | 0 | 2.0 | 2.8 | 1.4 | 3.1 | 3.1 | 1.0 | 10.9 | 9.2 | 0.8 | 17.8 | 13.1 | 0.7 | 20.0 | 14.2 | 0.7 | | | |
| No. 115 | 2690 | 4.2 | 13.6 | 13.0 | 1.0 | 15.8 | 18.8 | 1.2 | | | | | | | | | | | | |
| No. 117 | 2230 | 4.6 | 13.2 | 13.2 | 1.0 | 15.4 | 13.7 | 0.9 | | | | | | | | | | | | |
| No. 125 | 1840 | 6.0 | 11.8 | 10.3 | 0.9 | 14.0 | 12.0 | 0.9 | | | | | | | | | | | | |
| No. 127 | 2540 | 7.8 | 10.0 | 10.8 | 1.1 | 12.2 | 11.0 | 0.9 | | | | | | | | | | | | |
| No. 135 | 2560 | 9.5 | 8.3 | 7.8 | 0.9 | 10.5 | 7.2 | 0.7 | | | | | | | | | | | | |
| No. 138 | 1960 | 8.8 | 9.0 | 8.2 | 0.9 | 11.2 | 9.2 | 0.8 | | | | | | | | | | | | |
| No. 139 | 1920 | 8.0 | 9.8 | 9.6 | 1.0 | 12.0 | 10.4 | 0.9 | | | | | | | | | | | | |
| No. 148 | 2550 | 10.0 | 7.8 | 9.2 | 1.2 | 10.0 | 12.9 | 1.3 | | | | | | | | | | | | |
| No. 149 | 1910 | 9.0 | 8.8 | 3.9 | 0.4 | 11.0 | 12.8 | 1.2 | | | | | | | | | | | | |

* Uncoated wires. All other wires were zinc coated.

[a] Low copper content wire, 0.05 to 0.08 per cent Cu. All other wires have approximately 0.25 per cent Cu.

[b] Summarizes data through 1956.

The test results from specimens removed during the 1955 and 1956 inspections (Table B) are included in Tables VI through XI. These tables contain all the data accumulated to date for the bare and galvanized steel wires at Ames, Iowa, Manhattan, Kans., College Station, Tex., Davis and Santa Cruz, Calif., and Lafayette, Ind., respectively.

On the basis of tests to date, the loss in tensile strength by uncoated and zinc-coated steel wires after the wires begin to rust is shown in Table C.

Respectfully submitted on behalf of the Wire Test Inspection Committee,

FRED M. REINHART,
*Chairman.*

# REPORT OF SUBCOMMITTEE XVI

## ON

## FIELD TESTS OF ATMOSPHERIC CORROSION OF HARDWARE

This report covers the inspections made in 1955, 1956, and 1957 at the only remaining test site at State College, Pa. The data from these three inspections are shown in Table I. The last report of the discontinued test sites appear in the following *Proceedings* of the American Society for Testing Materials:

Altoona and Brunot Island, Pa.—1944 (Vol. 44),

Sandy Hook, N. J.—1952 (Vol. 52), and

Key West, Fla.—1952 (Vol. 52).

Progress of corrosion at State College continues at a very slow pace. No sign of deterioration is as yet noticeable on the aluminum-coated specimens after 28 years exposure. Hot-dip galvanized articles for the most part show only traces of yellow rust, although in a few cases as much as 80 per cent yellow rust and 20 per cent red rust have been recorded. Electro-galvanized samples having coating weights of 1.50 oz per sq ft or greater compare favorably with hot-dip galvanized coatings of comparable weights. Lead coatings are by and large considerably inferior to either zinc or aluminum in this atmosphere.

Respectfully submitted on behalf of the subcommittee,

A. Mendizza,
*Chairman.*

## TABLE I.—REPORT OF INSPECTION ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC., EXPOSED TO ATMOSPHERE ON TEST RACKS AT STATE COLLEGE, PA.

Abbreviations and symbols used: 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; Y, yellow or orange rust; R*, rust spots widely distributed; Tr, trace; TH, threads; C, average of nipple and ell.

| Coating | Weight, oz per sq ft of surface | 4-19-55<br>1353 weeks | 4-27-56<br>1406 weeks | 4-24-57<br>1458 weeks |
|---|---|---|---|---|
| 3 by 3 by $\frac{5}{16}$-in. Plain Steel Angles | | | | |
| Hot-dip galvanized | 2.52 | TrY | TrY | TrY |
| Hot-dip aluminum | 0.89 | 0 | 0 | 0 |
| Electric sherardized | 1.38 | TrR–100Y | 5R–95Y | 5R–95Y |
| Lead-plated (hot-dip) | ... | 5R–80R* | 10R–75R* | 25R–60R* |
| 3 by 3 by $\frac{5}{16}$-in. Copper-Bearing Angles | | | | |
| Hot-dip galvanized | 2.88 | TrY | TrY | TrR–TrY |
| Hot-dip aluminum | 0.82 | 0 | 0 | 0 |
| Electric sherardized | 1.44 | 15R–90Y | 20R–80Y | 25R–75Y |
| Lead-plated (hot-dip) | ... | 5R–85R* | 5R–80R* | 5R–75R* |
| $2\frac{1}{2}$ by $1\frac{1}{2}$ by $\frac{3}{16}$-in. Plain Steel Angles | | | | |
| Hot-dip galvanized | 3.24 | TrY | TrY | TrY |
| Hot-dip aluminum | 0.75 | 0 | 0 | 0 |
| Lead-plated (hot-dip) | ... | 10R–60R* | TrR–75R* | 10R–65R* |
| $2\frac{1}{2}$ by $1\frac{1}{2}$ by $\frac{3}{16}$-in. Copper-Bearing Angles | | | | |
| Hot-dip galvanized | 2.75 | TrY | TrY | TrY |
| Hot-dip aluminum | 0.50 | 0 | 0 | 0 |
| Lead-plated (hot-dip) | ... | 10R–85R* | 5R–80R* | 30R–65R* |
| 2-in. Diameter Tubular Goods | | | | |
| Electrogalvanized | 1.58 | 0 | 0 | TrR |
| Hot-dip galvanized | 2.51 | 15Y | 15Y | 25Y |
| Hot-dip aluminum | 4.73 | 0 | 0 | 0 |
| Gas sherardized | 0.66 | 50R–50Y | 55R–45Y | 60R–40Y |
| Lead-plated (hot-dip) | ... | 45R–55R* | 45R–55R* | 60R–40R* |
| 1-in. Tubular Goods | | | | |
| Electrogalvanized | 1.62 | 0 | 0 | 0 |
| Hot-dip galvanized | 1.83 | TrR–85Y | TrR–85Y | TrR–85Y |
| Hot-dip aluminum | 1.83 | 0 | 0 | 0 |
| Electric sherardized | 0.92 | 90R–10Y | 95R–5Y | 100R |
| Gas sherardized | 0.72 | 70R–30Y | 70R–30Y | 70R–30Y |
| Calorized | ... | 100Y | 5R*–95Y | 5R*–95Y |
| Lead-plated (hot-dip) | ... | 25R–75R* | 25R–75R* | 50R–50R* |
| Amaloy | ... | 15R–85R* | 10R–80R* | 10R–80R* |
| Forged Steel Tower Fittings | | | | |
| Hot-dip galvanized | 2.67 | 55Y | 70Y | 70Y |
| Hot-dip galvanized | 1.53 | 5R–90Y | 5R–95Y | 20R–80Y |
| Hot-dip aluminum | ... | 0 | 0 | 0 |
| Electric sherardized | 0.50 | 65R–35Y | 65R–35Y | 65R–35Y |

**(Continued on p. 132.)**

TABLE I.—(*Continued*)

| Coating | Weight, oz per sq ft of surface | 4-19-55 1353 weeks | 4-27-56 1406 weeks | 4-24-57 1458 weeks |
|---|---|---|---|---|
| ¾-IN. NIPPLES AND ELLS | | | | |
| Electrogalvanized MI[a] | 1.60 | 5R(C) | 10R(C) | 10R(C) |
| Electrogalvanized CI[b] | 2.19 | 20R–10Y(C) | 55R–10Y(C) | 20R–10Y(C) |
| Electrogalvanized MI | 2.65 | 60R–25Y(C) | 70R–30Y(C) | 70R–20Y(C) |
| Electrogalvanized CI | 1.25 | 40R–30Y(C) | 50R | 40R–20Y(C) |
| Hot-dip galvanized MI | 1.82 | TrR–100Y | TrR–80Y | 10R–80Y |
| Hot-dip galvanized CI | 1.44 | TrR–100Y | TrR–80Y | 10R–80Y |
| Hot-dip aluminum MI | 3.20 | RTH | RTH | RTH |
| Hot-dip aluminum CI | 2.80 | RTH | RTH | RTH |
| Electric sherardized MI | 0.75 | 25R–75Y(C) | 30R–70Y | 30R–70Y |
| Amaloy MI and CI | ... | 10R–90R* | 10R–90R* | 50R–50R* |
| TOWER CLAMPS | | | | |
| Hot-dip galvanized MI | 2.00 | 15R–85Y | 15R–90Y | 10R–90Y |
| Hot-dip galvanized CI | 2.38 | TrR–15Y | TrR–25Y | TrR–15Y |
| Hot-dip aluminum MI | 3.16 | 0 | 0 | 0 |
| Hot-dip aluminum CI | 2.51 | 0 | 0 | 0 |
| 1½ BY 1½ BY ⅛-IN. PLAIN STEEL ANGLES | | | | |
| Hot-dip galvanized | 3.16 | TrY | TrY | TrY |
| Hot-dip aluminum | 0.89 | 0 | 0 | 0 |
| Lead plated (hot-dip) | ... | 10R–60R* | 10R–60R* | 10R–60R* |
| 1½ BY 1½ BY ⅛-IN. COPPER-BEARING ANGLES | | | | |
| Hot-dip galvanized | 3.20 | TrY | TrY | TrY |
| Hot-dip aluminum | 1.11 | 0 | 0 | 0 |
| Lead-plated (hot-dip) | ... | 10R–85R* | 5R–85R* | 15R–75R* |
| NO. 14 BY 2½-IN. COPPER-BEARING STEEL FLATS | | | | |
| Hot-dip galvanized | 3.20 | TrR–TrY | TrR–TrY | TrR–TrY |
| Hot-dip aluminum | 0.47 | 0 | 0 | 0 |
| Lead plated (hot-dip) | ... | TrR–55R* | TrR–60R* | 20R–60R* |
| KNOCKOUT BOXES | | | | |
| Hot-dip galvanized | 1.50 | TrR–40Y | TrR–40Y | TrR–70Y |
| Electric sherardized | 0.28 | TrR–100Y | TrR–100Y | TrR–100Y |
| Amaloy | ... | 10R–85R* | 10R–85R* | 50R–50R* |
| Hot-dip aluminum | 1.84 | 0 | 0 | 0 |
| BULLDOG CLAMPS | | | | |
| Hot-dip aluminum | 1.35 | 0 | 0 | 0 |
| ¼ BY 3-IN. PLAIN STEEL FLATS | | | | |
| Hot-dip galvanized | 2.49 | TrY | TrY | TrY |
| Hot-dip aluminum | 0.90 | 0 | 0 | 0 |
| Lead-plated (hot-dip) | ... | TrR–90R* | TrR–90R* | 10R–85R* |

**(Concluded on p. 133.)**

TABLE I.—*Concluded*

| Coating | Weight, oz per sq ft of surface | 4-19-55<br>1353 weeks | 4-27-56<br>1406 weeks | 4-24-57<br>1458 weeks |
|---|---|---|---|---|
| ¼ by 3-in. Copper-Bearing Flats | | | | |
| Electrogalvanized | 0.84 | 90R-10Y | 95R-5Y | 95R-5Y |
| Hot-dip galvanized | 2.67 | TrY | TrY | TrY |
| Hot-dip aluminum | 0.66 | 0 | 0 | 0 |
| Lead-plated (hot-dip) | ... | TrR-75R* | TrR-75R* | 5R-85R* |
| No. 792 Window Frame Section | | | | |
| Hot-dip galvanized | 2.60 | TrY | TrY | TrY |
| Hot-dip aluminum | 1.39 | 0 | 0 | 0 |

[a] MI = malleable iron.

[b] CI = cast iron.

# REPORT OF COMMITTEE A-9
ON
# FERRO-ALLOYS*

Committee A-9 on Ferro-Alloys held one meeting during the year: in Atlantic City, N. J. on June 20, 1956.

The committee consists of 25 members, of whom 10 are classified as producers, 9 as consumers, and 6 as general interest members.

## REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Ferromanganese (**A 99 – 53 T**[1] and continuation of the specifications as tentative:

*Table I.*—For standard ferromanganese change the manganese content for grade B from "74.0 to 78.0" to read "76.0 to 78.0," and add a new grade C with a manganese content of 74.0 to 76.0, the requirements for the other elements to be the same as for grades A and B.

For all grades of medium-carbon and low-carbon ferromanganese, change the manganese content from "80.0 to 85.0" to read "80.0" per cent, minimum.

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 25 members; 22 members returned ballots, of whom 21 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. W. POOLE,
*Chairman.*

W. H. MAYO,
*Secretary.*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 1.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE A-10

## ON

# IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS*

Committee A-10 on Iron-Chromium, Iron - Chromium - Nickel and Related Alloys held two meetings during the year: in Atlantic City, N. J. in June, 1956, during the Annual Meeting of the Society, and in February, 1957, at Philadelphia, Pa., during ASTM Committee Week.

The committee consists of 103 members, of whom 45 are classified as producers, 39 as consumers and 19 as general interest members.

M. A. Cordovi resigned as secretary of the committee, and G. N. Emmanuel was appointed secretary to fill out the unexpired portion of the term.

## NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Corrosion-Resisting Chromium-Nickel-Manganese Steel (Types 201 and 202) Plate, Sheet and Strip as appended hereto.[1]

## REVISIONS OF STANDARDS AND TENTATIVES

As indicated in the report of the Joint A-1, A-10 Subcommittee on Stainless Tubing Specifications, a number of revisions of specifications for stainless tubing materials have been agreed upon in the joint committee. These are set forth in the Annual Report of Committee A-1 and the individual recommendations are given in that report. The recommendations affect the following five specifications under the joint jurisdiction of Committees A-1 and A-10:

*Standard Specifications for:*

Seamless and Welded Ferritic Stainless Steel Tubing for General Service (**A 268 - 55**),
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (**A 269 - 56**),
Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (**A 270 - 55**), and
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (**A 271 - 56**).

*Tentative Specifications for:*

Seamless and Welded Austenitic Stainless Steel Pipe (**A 312 - 56 T**).

These are now receiving consideration in Subcommittee XI of Committee A-10 looking toward securing action on the part of that subcommittee and of Committee A-10.

## ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Recommended Practice for Descaling and Cleaning Stainless Steel Surfaces (**A 380 - 54 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without change.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee IV on Methods of Cor-*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 1

[2] 1955 Book of ASTM Standards, Part 1.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*rosion Testing* (O. B. Ellis, chairman) recommended the adoption as standard of the Tentative Recommended Practice for Descaling and Cleaning Stainless Steel Surfaces (A 380) referred to earlier in the report.

It is planned to expose samples of stainless steel for atmospheric tests in the spring of 1958 at State College, Pa., Kure Beach, N. C., Freeport, Tex. and New York City area.

The task group on stress corrosion cracking of austenitic stainless steel (F. K. Bloom, chairman) is studying the data received in response to the questionnaires which had been circulated and a report will be issued when the initial studies have been completed.

Special stainless steel specimens exposed on the roof of the New York Port Authority Building were moved to the new ASTM Test Site near Newark, N. J. A spring inspection is planned of these specimens as well as those exposed at Pittsburgh, Pa.

*Subcommittee V on Mechanical Testing* (R. H. Heyer, chairman) has approved a test specimen modification for tension testing of stainless steel plate in the thicknesses between $\frac{1}{2}$ in. and $\frac{5}{8}$ in. The maximum allowable thickness of the present 2-in. gage length flat tension specimen of Methods E 8, Fig. 6, would be increased from $\frac{1}{2}$ in. to $\frac{5}{8}$ in. in tests on stainless steels. If the change in the 2-in. gage length specimen is approved, the present 8-in. gage length flat tension specimen will be removed from the specifications covering stainless steel plate, and the modified 2-in. gage length specimen added.

Task groups are preparing new recommendations for speed of testing of the austenitic grades, and obtaining data for the establishment of hardness conversion tables for stainless steels.

*Subcommittee VI on Metallography* (R. Franks, chairman).—A task group is working on metallographic studies to identify constituents of heavy-section welded type 347 stainless steels that have caused difficulties due to cracking. A number of different samples have been examined, some of which have failed in service while others have performed satisfactorily. It is anticipated that some clue as to the identity of the brittle constituent will be obtained.

It is planned to set up another task group to complete the work done some time ago on the identification of the sigma phase in small particle size in stainless steels, particularly in the chromium-nickel types of stainless steels with and without molybdenum.

*Subcommittee VIII on Specifications for Wrought Products* (R. B. Gunia, chairman).—The task group appointed to write a standard covering AISI types 201 and 202 has prepared a rough draft which will be submitted to the main committee some time in 1957.

*Subcommittee IX on Specifications for Flat Products* (G. W. Hinkle, chairman) prepared the new specification for corrosion-resisting chromium-nickel-manganese steel plate, sheet and strip referred to earlier in the report.

Revisions of Specifications A 240 are being considered relating to check analysis, gage sizes, and weights.

A task force was appointed to consult with a similar group from Subcommittee XI of Committee A-1 to establish procedures or tests for heat treatment of stainless steel clad materials.

*Subcommittee X on Specifications for Castings* (J. J. Kanter, chairman) is considering revisions concerning the mechanical properties and chemical composition of grades HH, HI, HK, CB-30 and CB-50 in Specifications A 296 and A 297. It is contemplated that a new grade, CA-40 will be added to Specifications A 296.

*Subcommittee XIII on Specifications for Nuclear Reactor Materials* (M. A. Cordovi, chairman) is to review specifications for all materials which are used or may be used in nuclear applications under the jurisdiction of Committee A-10 and recommend changes or new specifications as necessary. Final approval of any recommendations rest with the main committee.

It has been recommended to Subcommittee IX that there is a need for a modified type 430 material in Specifications A 268. The general and stress-corrosion characteristics of this material were listed.

*Joint A-1, A-10 Subcommittee on Stainless Tubing Specifications* (J. J. B. Rutherford, chairman) has approved the following recommendations which in turn were approved by Subcommittee IX of Committee A-1 and are included in the A-1 Report: new Tentative Specifications for Welded Large Outside Diameter Light Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service; increase in the phosphorus content to 0.040 per cent, max, of all stainless steel grades in Specifications A 268, A 269, A 270, A 271 and A 312; and addition of grades TP 304 L and TP 316 L to Specifications A 269 and A 312.

Other items agreed upon affect only standards under the sole jurisdiction of Committee A-1.

This report has been submitted to letter ballot of the committee, which consists of 103 members; 74 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
*Chairman.*

G. N. EMMANUEL,
*Secretary.*

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EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Revision and Reversion to Tentative of Standard Specifications for:*

Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 – 54), and
Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (A 276 – 55).

*Withdrawal of Emergency Alternate Provisions for:*

Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet and Strip for Fusion-Welded Unfired Pressure Vessels (EA – A 240), and
Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (EA – A 276).

The revised specifications appear in the 1957 Supplement to Book of ASTM Standards, Part 1.

# REPORT OF COMMITTEE B-1

## ON

## WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: in Washington, D. C., on October 23, 1956; in New York, N. Y., on January 29, 1957; and in Pittsburgh Pa., on March 29, 1957. Meetings of Subcommittees II, IV, and VII also were held during the year.

The committee notes with regret the retirement on July 1, 1956, of S. Skowronski, a long-time member of the committee who contributed much to its work, and the retirement on June 29, 1956, of P. V. Faragher who served as chairman of Subcommittee VII for ten years. Mr. Faragher will continue his committee membership on a personal basis.

The vacancy in the chairmanship of Subcommittee VII has been filled by A. L. Kolb.

The committee consists of 87 members, of whom 44 are classified as producers, 28 as consumers, and 15 as general interest members.

### NEW TENTATIVE

The committee recommends for publication as tentative the proposed Tentative Specifications for Aluminum Wire for Communication Cable, as appended hereto.[1]

With the increasing availability of electrical conductor grade aluminum and the shortage of copper in recent years, the application of aluminum to conductor uses other than bare overhead transmission has been growing. The committee has recognized this trend and has been developing proposed specifications for several particular types of aluminum conductors.

### REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following two specifications and their continuation as tentative:

**Tentative Specifications for Concentric-Lay-Stranded Aluminum Conductors, Steel Reinforced (ACSR) (B 232 - 56 T):**[2]

*Section 5.*—Change Paragraphs *(g)* and *(h)* to read as follows:

(*g*) The preferred lay of the 6 wire layers of 7 and 19 wire steel cores is 25 times the outside diameter of the 6 wire layer but shall be not less than 18 nor more than 30 times that diameter.

(*h*) The preferred lay of the 12 wire layer of a 19 wire steel core is 20 times the outside diameter of the core but shall be not less than 16 nor more than 24 times that diameter.

Delete Paragraph *(i)* and reletter Paragraph *(j)* as *(i)*.

**Tentative Specifications for Soft or Annealed Coated Copper Conductors for Use in Hookup Wire for Electronic Equipment (B 286 - 54 T):**[3]

*Section 2.*—Revise Items *(1)*, *(2)*, and *(3)* to read as follows:

(*1*) Quantity of each size designation (Table II) and type,

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 2.

[2] 1956 Supplement to Book of ASTM Standards, Part 2.

[3] 1955 Book of ASTM Standards, Part 2.

## TABLE I.—DETAILS OF CONDUCTOR CONSTRUCTION.

Revision of Table II, Specifications B 286 – 54 T)

### TYPE I (SOLID CONDUCTORS)

| Size Designation, Awg | Nominal Area, cir mils | Diameter, in. | | | D-c Resistance at 20 C, ohms per 1000 ft, max (Note 1) |
|---|---|---|---|---|---|
| | | Nominal | Minimum | Maximum | |
| 10 | 10380 | 0.1019 | 0.1009 | 0.1050 | 1.06 |
| 12 | 6530 | 0.0808 | 0.0800 | 0.0832 | 1.69 |
| 14 | 4110 | 0.0641 | 0.0635 | 0.0660 | 2.68 |
| 16 | 2580 | 0.0508 | 0.0503 | 0.0523 | 4.26 |
| 18 | 1620 | 0.0403 | 0.0399 | 0.0415 | 6.78 |
| 20 | 1020 | 0.0320 | 0.0317 | 0.0330 | 10.8 |
| 22 | 640 | 0.0253 | 0.0251 | 0.0261 | 17.2 |
| 24 | 404 | 0.0201 | 0.0199 | 0.0207 | 27.2 |
| 26 | 253 | 0.0159 | 0.0157 | 0.0164 | 44.5 |

### TYPE II (STRANDED CONDUCTORS)

| Size Designation | Conductor Construction | | Calculated Cross-Sectional Area, cir mils | Theoretical Average Diameter, in.[b] | Length of Lay, in. (Note 2) | D-c Resistance at 20 C, ohms per 1000 ft, max (Note 1) |
|---|---|---|---|---|---|---|
| | Number of Wires[a] | Nominal Diameter of Each Wire, in. | | | | |
| 0000-2109 | 2109 | 0.0100 | 210900 | 0.624 | ... | 0.0577 |
| 000-1672 | 1672 | 0.0100 | 167200 | 0.556 | ... | 0.0728 |
| 00-1330 | 1330 | 0.0100 | 133000 | 0.496 | ... | 0.0915 |
| 0-1064 | 1064 | 0.0100 | 106400 | 0.443 | ... | 0.114 |
| 1-836 | 836 | 0.0100 | 83600 | 0.393 | ... | 0.146 |
| 2-665 | 665 | 0.0100 | 66500 | 0.346 | ... | 0.181 |
| 4-420 | 420 | 0.0100 | 42000 | 0.275 | ... | 0.287 |
| 6-266 | 266 | 0.0100 | 26600 | 0.219 | ... | 0.453 |
| 8-168 | 168 | 0.0100 | 16800 | 0.174 | ... | 0.717 |
| 10-104 | 104 | 0.0100 | 10400 | 0.121 | 1.7 to 2.1 | 1.14 |
| 12-65 | 65 | 0.0100 | 6500 | 0.096 | 1.3 to 1.7 | 1.82 |
| 12-19[c] | 19 | 0.0179 | 6088[c] | 0.091 | 1.05 to 1.45 | 1.92 |
| 14-41 | 41 | 0.0100 | 4100 | 0.076 | 1.15 to 1.35 | 2.88 |
| 14-19[c] | 19 | 0.0142 | 3831[c] | 0.072 | 0.80 to 1.15 | 3.08 |
| 16-26 | 26 | 0.0100 | 2600 | 0.060 | 0.60 to 0.90 | 4.55 |
| 16-19[c] | 19 | 0.0113 | 2426[c] | 0.058 | 0.60 to 0.90 | 4.87 |
| 18-26[c] | 26 | 0.0080 | 1664[c] | 0.047 | 0.50 to 0.70 | 7.10 |
| 18-19[c] | 19 | 0.0100 | 1900[c] | 0.051 | 0.50 to 0.70 | 6.16 |
| 18-7[c] | 7 | 0.0159 | 1770[c] | 0.049 | 0.50 to 0.70 | 6.61 |
| 20-19[c] | 19 | 0.0080 | 1216[c] | 0.041 | 0.45 to 0.55 | 9.62 |
| 20-10 | 10 | 0.0100 | 1000 | 0.038 | 0.45 to 0.55 | 11.8 |
| 20-7[c] | 7 | 0.0126 | 1111[c] | 0.038 | 0.45 to 0.55 | 10.52 |
| 22-19[c] | 19 | 0.0063 | 754[c] | 0.032 | 0.33 to 0.43 | 15.5 |
| 22-7[c] | 7 | 0.0100 | 700[c] | 0.031 | 0.33 to 0.43 | 16.9 |
| 24-19[c] | 19 | 0.0050 | 475[c] | 0.026 | 0.25 to 0.35 | 24.6 |
| 24-7[c] | 7 | 0.0080 | 448[c] | 0.025 | 0.25 to 0.35 | 26.4 |
| 26-7[c] | 7 | 0.0063 | 278[c] | 0.020 | 0.20 to 0.30 | 42.5 |
| 28-7[c] | 7 | 0.0050 | 175[c] | 0.016 | 0.20 to 0.30 | 67.5 |
| 30-7[c] | 7 | 0.0040 | 112[c] | 0.013 | 0.20 to 0.30 | 105.5 |
| 32-7[c] | 7 | 0.0031 | 67[c] | 0.010 | 0.20 to 0.30 | 176.0 |

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[a] The stranded conductor constructions shown in this table provide for finished noninsulated conductors having the indicated cross-sectional area. The number of component wires may vary slightly provided the specified resistances are not exceeded.

[b] The theoretical average diameters of these conductors are given here for guidance in making calculations regarding insulating material, etc. These diameters do not include allowance for distortion of the conductor during stranding and are not intended to be used as limiting values.

[c] The cross-sectional areas of these conductor size designations deviate by more than 2 per cent from the nominal areas of the standard Awg sizes as defined in ASTM Specifications B 258.

(*2*) Conductor size designation, construction, and type (Table II),

(*3*) Desired constructions where alternates are given (Table II, Type II, Sections 4(*b*), 5(*a*), and 5(*b*)).

TABLE II.—D-C RESISTANCE AT 20 C FOR FINISHED INSULATED CONDUCTORS.

(Revision of Table III, Specifications B 286)

TYPE I (SOLID CONDUCTORS)

| Size Designation, Awg | Resistance at 20 C, ohms per 1000 ft, max | Size Designation, Awg | Resistance at 20 C, ohms per 1000 ft, max |
|---|---|---|---|
| 10 | 1.10 | 20 | 11.2 |
| 12 | 1.76 | 22 | 17.9 |
| 14 | 2.79 | 24 | 28.3 |
| 16 | 4.44 | 26 | 46.3 |
| 18 | 7.05 | | |

TYPE II (STRANDED CONDUCTORS)

| Size Designation | Resistance at 20 C, ohms per 1000 ft, max | Size Designation | Resistance at 20 C, ohms per 1000 ft, max |
|---|---|---|---|
| 0000-2109 | 0.0588 | 18-26 | 7.39 |
| 000-1672 | 0.0742 | 18-19 | 6.41 |
| 00-1330 | 0.0933 | 18-7 | 6.87 |
| 0-1064 | 0.116 | 20-19 | 10.0 |
| 1-836 | 0.149 | 20-10 | 12.3 |
| 2-665 | 0.185 | 20-7 | 10.9 |
| 4-420 | 0.293 | 22-19 | 16.1 |
| 6-266 | 0.462 | 22-7 | 17.6 |
| 8-168 | 0.731 | 24-19 | 25.6 |
| 10-104 | 1.16 | 24-7 | 27.5 |
| 12-65 | 1.86 | 26-7 | 44.2 |
| 12-19 | 2.00 | 28-7 | 70.2 |
| 14-41 | 2.94 | 30-7 | 109.7 |
| 14-19 | 3.20 | 32-7 | 183.0 |
| 16-26 | 4.73 | | |
| 16-19 | 5.06 | | |

*Section 4*(*b*).—Change to read as follows:

(*b*) Stranded conductors shall conform to the requirements of type II conductors prescribed in Table II. The method of stranding for conductor size designation 32-7 through 10-104 inclusive, shall be at the option of the manufacturer unless otherwise specified. Stranded conductors size designation 8-168 and larger shall normally be furnished in a rope-lay-stranded construction consisting of either 7 or 19 bunch-stranded members.

*Section 5.*—In Paragraph (*a*), change the first sentence to read "The direction of lay of the outside layer of stranded conductors shall be left hand." Change Paragraph (*c*) to read as follows:

(*c*) The length of lay of the outside layer of stranded conductors in size designation 32-7 through 10-104, inclusive, shall conform to the values in Table II (Note 2). For strand constructions containing more than one distinct layer the length of lay of the inner layer shall not exceed the maximum value shown in Table II for the conductor in question. For rope-lay-stranded conductors size designation 8-168 and larger, the length of lay of the wires composing the bunch-stranded members shall be not more than 30 times the diameter of the member, and the length of lay of the outer layer of rope-lay-stranded conductors shall be not less than 8 nor more than 16 times the outside diameter of the completed conductor.

*Section 6.*—In the first sentence change "No. 30 Awg" to read "size designation 32-7." and change "No. 10 Awg" to read "size designation 10-104." Replace the period at the end of the second sentence with the word "and."

*Table II.*—Change Table II to read as shown in the accompanying Table I.

*Table III.*—Change Table III to read as shown in the accompanying Table II.

*Section 10.*—In the first sentence change "size" to read "size designation."

*Explanatory Notes.*—Add at the end of Note 1, a new paragraph to read as follows:

The method used to calculate the resistance values appearing in Tables II and III is shown below:

*D-C Resistance at 20 C for the Bare Conductors* (*Table II*):

$$\text{ohms per 1000 ft} = \frac{\rho \times 105.35 \times f \times m}{d^2 \times \delta \times N \times 0.98}$$

where:

$\rho$ = 910.15 ohms (pound/mile$^2$) for wires 0.0201 in. and larger,

929.52 ohms (pound/mile$^2$) for wires 0.0111 in. incl. to 0.0201 in.,
939.51 ohms (pound/mile$^2$) for wires under 0.0111 in. (See Table II, ASTM Specifications B 33 and B 189)

$d$ = wire diameter in mils,
$\delta$ = density of the wire (8.89 g per cu cm),
$N$ = number of wires composing conductor,
$f$ = stranding lay factor,
1.00 for single wire conductors,
1.02 for bunch-stranded conductors,
1.04 for rope-lay-stranded conductors containing 7 bunched members. (See ASTM Specifications B 172, Note 8). Conductors in sizes No. 8 Awg through No. 2 Awg, incl, are assumed to be made up of 7 bunched members.
1.05 for rope-lay-stranded conductors containing 19 bunched members. Conductors larger than No. 2 Awg are assumed to be made up of 19 bunched members.
$m$ = Stretch allowance factor to compensate for stretching of the conductor during stranding. For solid conductors, the value of *m* therefore is 1.00, but for bunch stranded and rope-lay-stranded conductors *m* is taken as 1.02,
0.98 in the denominator of the foregoing equation is the cross-section area factor derived from the wire diameter *minus* tolerance permitted in both ASTM Specifications B 33 and B 189.

*D-C Resistance at 20 C of the Insulated Conductors (Table III).*

where: ohms per 1000 ft = resistance before insulation multiplied by 1.04
1.04 = Stretch allowance factor to compensate for stretch of the conductor while being insulated.

## TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire Fabrication (**B 279 – 53 T**).

## ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specifications for Standard Nominal Diameters and Cross-Sectional Areas of Awg Sizes of Solid Round Wires Used as Electrical Conductors (**B 258 – 51 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated. This specification has stood for six years as tentative, during which time only minor editorial changes have been made, and no criticisms or suggestions for improvement of a material nature have been received.

*Table II.*—Change heading of last column to read, "Resistivity, $\rho$, at 20 C, ohm—pound/mile$^2$."

*Section 7.*—Change "D-c resistance at 20 C, feet per ohm" to read "Length at 20 C, feet per ohm." Change "D-c resistance at 20 C, lb per ohm" to read "Weight at 20 C, lb per ohm." Change "$\rho$ = resistivity of the wire material at 20 C in ohms (mile, pound)" to read "$\rho$ = resistivity of the wire material at 20 C in ohm—pound/mile$^2$."

*Explanatory Notes.*—In Note 2, in the first sentence, change "ohms (mile, pound)" to read "ohm—pound/mile$^2$" and in the third sentence change "ohm (meter, gram)" to read "ohm—gram/meter$^2$."

*Table III.*—In the first column, and in all column headings, make changes in unit designations as follows:

| Present | Proposed |
|---|---|
| ohms (mil, foot) | ohm—circular mil/ft |
| ohms (meter, square millimeter) | ohm—mm$^2$/meter |
| microhms—inch | microhm—inch |
| microhms—centimeter | microhm—cm |
| ohms (mile, pound) | ohm—pound/mile$^2$ |
| ohms (meter, gram) | ohm—gram/meter$^2$ |

## REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following four standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

### Standard Specifications for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (B 48 - 55):[3]

*Section 1(b).*—Add the following as the fourth and fifth paragraphs:

*Type L.*—For applications involving winding operations where minimum stiffness is required.

*Type BL.*—For applications involving both edgewise bending and winding operations for which the requirements for both types B and L will apply.

*Section 7.*—Change Paragraph (*b*) to read, "For types B, L, and BL wire the tensile and elongation tests will not be required." Delete Paragraph (*d*).

*Section 8(a).*—In the first sentence change "Type B" to read "Types B and BL." In last sentence, change "class B" to read "types B and BL."

*Section 8(b).*—Change to read, "For types A and L wire the bend test will not be required."

*New Sections.*—Add the following new Sections 9 and 10, renumbering the subsequent sections accordingly:

9. *Low Stress Elongation (LSE) Properties.*—(*a*) Types L and BL wire shall have a minimum LSE value of 1 per cent determined in accordance with the Method of Test for Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire Fabrication (ASTM Designation: B 279) (Note 12).

(*b*) For types A and B wire the LSE test will not be required.

10. *Retests.*—If upon testing a sample from any coil or reel of wire the results do not conform to the respective requirements of Section 7, 8, or 9, two additional samples shall be tested, each of which shall conform to the prescribed requirements.

*New Note 12.*—Add a new Note 12 to read as follows:

NOTE 12.—LSE test results are affected by small amounts of cold working. The specified minimum 1 per cent LSE value applies only to bare wire before further processing.

### Standard Method of Test for Resistivity of Electrical Conductor Materials (B 193 - 49):[3]

*Section 2(b).*—In the first paragraph and in the formula, change "ohms (mil, foot)" and "ohms (meter, square millimeter)" to read respectively "ohm—circular mil/ft" and "ohm—mm$^2$/meter."

*Note 1.*—Change "ohms (mil, foot)" and "ohm (meter, square millimeter)" to read respectively "ohm—circular mil/ft" and "ohm—mm$^2$/meter."

*Table I.*—Change unit designations to read as recommended above for Table III of Specifications B 258.

*Note 2.*—In the first paragraph and in the calculation, change "ohms (mile, pound)" and "ohms (meter, gram)" to read respectively "ohm—pound/mile$^2$" and "ohm—gram/meter$^2$."

*Table II.*—Replace the listing for "Aluminum, Grade EC" with the following:

| Material | Density | Temperature Coefficient |
|---|---|---|
| Aluminum, grade EC, per cent IACS: | | |
| 61.8 | 2.703 | 0.00408 |
| 61.5 | 2.703 | 0.00406 |
| 61.4 | 2.703 | 0.00406 |
| 61.3 | 2.703 | 0.00405 |
| 61.0 | 2.703 | 0.00403 |

*Table III.*—Change unit designations to read as recommended above for Table III of Specifications B 258.

### Standard Specifications for Hard-Drawn Copper Covered Steel Wire (B 227 - 52):[3]

*Table I.*—For grade 30 EHS of 0.0800 in. nominal diameter insert a minimum tensile strength of "179,000" psi.

*Table III.*—For grades 30 HS and 30 EHS of under 0.1000 to 0.0800 in., incl, in diameter insert a minimum thickness of copper of "4" mils.

*Table IV.*—For grade 30 EHS of 0.0800 in. in nominal diameter insert a

minimum breaking strength of "900" psi. Also for grades 30 HS and 30 EHS of 0.0800 in. nominal diameter insert a resistance at 20 C of "5.509" ohms per 1000 ft.

### Standard Specifications for Concentric-Lay-Stranded Aluminum Conductors, Hard-Drawn and Three-Quarter Hard Drawn (B 231 – 55):[3]

*Section 7.*—Change the heading "Physical and Electrical Tests" to read, "Physical and Electrical Tests on Conductors with Hard-Drawn Aluminum Wires and Conductors Fabricated from Three-Quarter Hard-Drawn Aluminum Wires and NOT Annealed after Stranding."

*New Section.*—Add a new Section 8 to read as follows, renumbering the subsequent sections accordingly:

8. *Physical and Electrical Tests on Conductors Fabricated from Aluminum Wires other than Three-Quarter Hard-Drawn Temper AND Annealed after Stranding to Meet Three-Quarter Hard-Drawn Requirements.*—(*a*) Tests for the physical and electrical properties of wires removed from the completed conductor shall be made as specified in Paragraph (*b*). Upon agreement between the purchaser and the manufacturer, tests on wires removed from the completed conductor may be waived and the completed conductor tested as a unit as provided in Paragraph (*c*).

(*b*) The tests made on wires removed from the completed conductor shall have tensile strengths not less than 95 per cent of the minimum tensile strength nor more than 105 per cent of the maximum tensile strength prescribed in Section 5(*a*) of the Specifications for Three-Quarter Hard Aluminum Wire for Electrical Purposes (ASTM Designation: B 262).

(*c*) When the completed conductor is tested as a unit, the minimum breaking strength of three-quarter hard-drawn bare conductors shall be at least 90 per cent of the total of the calculated minimum breaking strengths of the component wires. The minimum breaking strengths of the component wires shall be calculated using specified nominal wire diameters and specified minimum tensile strengths for three-quarter hard-drawn conductors. The maximum breaking strength of conductors made from three-quarter hard wires shall be not greater than the sum of the calculated maximum breaking strengths of the component wires. The maximum breaking strengths of the component wires shall be calculated using specified nominal wire diameters and specified maximum tensile strengths. The free length between grips of the test specimen shall be not less than 24 in., and care shall be taken to insure that the wires in the conductor are evenly gripped during the test (Note 4).

(*d*) All wires composing the conductors shall be capable of meeting the bending properties stated in Specifications B 230 after stranding. Routine production testing after stranding is not required.

### EDITORIAL CHANGES

The committee recommends the following editorial changes in the Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 56):[2]

*Table II.*—Insert the italic "a" as a superscript adjacent to the number of strands, for sizes 1 to 4 Awg inclusive (3-wire conductors), listed in the third column (under Class AA, Number of Wires), e.g.:

| Size Awg | Class AA |
|---|---|
| | Number of Wires |
| 1 | 3[a] |
| 2 | 3[a] |
| 3 | 3[a] |
| 4 | 3[a] |

[a] Although class AA conductors having 3 strands do not conform to the construction requirements of Section 1 (*a*), they are listed in this table for convenience.

The committee also took action during the year to make editorial revisions as soon as practicable in all specifications under its jurisdiction to: (*a*) conform to National Bureau of Standards Circular 31, 4th Edition, wherever unit designations for resistivity appear, and (*b*) reference ASTM B 193 as the standard method of test, wherever resistivity requirements appear.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Editorial and Records* (A. A. Jones, chairman) continued its editorial review of specifications and coordinated the various subcommittee recommendations regarding revisions of specifications.

*Subcommittee II on Methods of Test and Sampling Procedures* (R. H. Lloyd, chairman) completed revision of Specifications B 193 and B 258 incorporating resistivity units conforming to NBS Circular 31, 4th Edition, and including reference values of density and temperature coefficient of resistance for several conductivities of grade EC aluminum. It is continuing to develop a method of calculating lay factor for bunch-stranded conductors.

*Subcommittee IV on Conductors of Copper and Copper Alloys* (B. J. Sirois, chairman) completed revision of Specifications B 48 to include low stress elongation (LSE) requirements, and of Specifications B 286 in cooperation with RETMA to include sizes and requirements having widespread usage in the electronic and aircraft industries.

Task Groups are cooperating with the U. S. Army Signal Corps Engineering Laboratories in the study of grey-tin transformation, considering revision of corner radii requirements in Specifications B 48 to permit improved quality of film-coated magnet wire, cooperating with Underwriters' Laboratories in an endeavor to coordinate length of lay requirements in small copper conductors, and investigating means for measuring wire diameters with a precision greater than 0.1 mil (that is, in hundredths of a mil) as a preliminary to establishing diameter tolerances for very fine wires.

*Subcommittee V on Conductors of Ferrous Metals* (L. H. Winkler, chairman) has a Task Group preparing a proposed specification for aluminum-coated steel core wire for ACSR constructions.

*Subcommittee VI on Composite Conductors of Copper and Steel* (C. E. Ambelang, chairman) completed revision of Specifications B 227 to provide for the inclusion of 0.0800 in. diameter, 30 per cent conductivity, extra high strength, hard-drawn copper-covered steel wire.

*Subcommittee VII on Conductors of Light Metals* (A. L. Kolb, chairman) completed revision of Specifications B 231 to cover the testing of completed stranded conductors having a temper different from that of the wire from which stranded ($\frac{3}{4}$-hard-drawn strand, made by stranding HD wires and subsequently partially annealing), and of Specifications B 232 to reconcile the lay length requirements for 7-wire cores with those for the 7-wire centers of 19-wire cores. The subcommittee also completed preparation of the proposed Tentative Specifications for Aluminum Wire for Communication Cable.

Task groups are considering the preparation of proposed specifications for aluminum strap for magnet wire, and for $\frac{1}{2}$-hard aluminum wire.

Joint Task Groups are considering the preparation of standard test report forms for bare electrical conductors, and a glossary of terms defining the various terms used in specifications under the jurisdiction of Committee B-1.

This report has been submitted to letter ballot of the committee, which consists of 87 members; $64\frac{1}{2}$ members returned their ballots, of whom $63\frac{1}{2}$ have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

D. HALLORAN,
*Chairman.*

A. A. JONES,
*Secretary.*

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE B-2

ON

# NON-FERROUS METALS*

Committee B-2 on Non-Ferrous Metals and Alloys met on June 20, 1956, in Atlantic City, N. J. A meeting of the Advisory Committee was held on April 18, 1957 in New York City.

A Symposium on Titanium sponsored by the committee at the Second Pacific Area National Meeting in September, 1956, in Los Angeles, Calif., has been issued as separate publication *ASTM STP No. 204.*

Subcommittee V on Precious Metals has had little activity for several years and has been discharged.

A new Subcommittee IX on Titanium and Titanium Alloys was formed with Thomas W. Lippert as chairman.

*Personnel.*—The committee consists of 130 members, of whom 54 are classified as producers, 51 as consumers, and 25 as general interest members.

A. M. Bounds, Superior Tube Co., succeeds W. A. Mudge as secretary of the committee.

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Refined Copper* (William E. Milligan, chairman) plans to compile specifications for three grades of phosphorized copper as requested by Committee B-5.

*Subcommittee II on Refined Lead, Tin, Antimony and Bismuth* (Sidney Rolle, chairman).—Task groups have prepared a Specification for Refined Secondary Lead, and a Tentative Recommended Practice for the Classification of Tin. The data collected as a basis for this latter document will be presented in a report to be published in the ASTM BULLETIN.

A request for specifications for sheet lead to be used in nuclear work will be explored.

*Subcommittee III on White Metal Alloys* (G. H. Clamer, chairman) is planning a further Symposium on Solder, the tentative date being June, 1960.

Studies are under way on procedures for the evaluation of soldered joints and procedures for the evaluation of fluxes. Revisions have been prepared in the Specifications for Soft Solder Metal (B 32 – 49) and Specifications for Rosin Flux Cored Solder (B 284 – 53 T), and upon approval by the Main Committee the revisions will be submitted to the Administrative Committee on Standards.

*Subcommittee IV on Refined Zinc and Wrought Zinc* (E. H. Bunce, chairman) has surveyed the desires of this subcommittee for participation in activities of the International Organization for Standardization (ISO) in establishing standards for zinc. Several producers have expressed interest in participating actively in this development.

*Subcommittee VII on Refined Nickel and Cobalt, High-Nickel Alloys and High-Cobalt Alloys Cast and Wrought* (Victor Franceschini, chairman; E. R. Patton, secretary).—A general revision of all the specifications covering nickel and nickel alloys is under way.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

*Subcommittee VIII on Miscellaneous Refined Metals and Alloys* (E. E. Schumacher, chairman).—As the result of task group surveys, work will begin immediately on standards for lithium and zirconium. The task groups for columbium, tantalum, thorium, uranium, beryllium, and hafnium will continue to appraise the situations and report next year. The task group for thallium will be disbanded. Associated subjects, such as standardization in the nuclear field, and effect of radiation on properties of metals will be followed by the subcommittee.

Task Groups to consider specifications for molybdenum and tungsten will be set up.

This report has been submitted to letter ballot of the committee, which consists of 130 members; 110 members returned their ballots, of whom 110 have voted affirmatively and 0 negatively.

Respectively submitted on behalf of the committee,

B. W. GONSER,
*Chairman.*

A. M. BOUNDS,
*Secretary.*

# REPORT OF COMMITTEE B-3

ON

# NON-FERROUS METALS AND ALLOYS*

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held two meetings during the year: in Atlantic City, N. J., on June 21, 1956, and in Philadelphia, Pa., on February 5, 1957.

The committee consists of 97 members, of whom 85 are voting members; 34 are classified as producers, 30 as consumers, and 19 as general interest members.

## REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

**Tentative Method for Salt Spray (Fog) Testing (B 117 – 54 T):**[1]

*Section 8 (b).*—In the third sentence change "0.50 to 3.0 ml." to read "0.75 to 2.0 ml."

**Tentative Method for Acetic Acid-Salt Spray (Fog) Testing (B 287 – 54 T):**[1]

*Section 6.*—In the third sentence change "3.2 to 3.5" to read "3.1 to 3.3." Add as the fourth sentence, "The pH must be measured on a sample of the collected spray."

*Section 8 (b).*—In the third sentence change "0.50 to 3.0 ml" to read "0.75 to 2.0 ml."

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following test methods be continued as tentative without change:

*Tentative Method of:*

Total Immersion Corrosion Test of Non-Ferrous Metals (**B 185 – 43 T**), and

Alternate Immersion Corrosion Test of Non-Ferrous Metals (**B 192 – 44 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee III on Spray Test* (C. O. Durbin, chairman).—On the basis of cooperative tests carried out by a task group, the subcommittee recommended the revisions of Methods B 117 and B 287 which are expected to make the tests more reproducible.

*Subcommittee VI on Atmospheric Corrosion* (H. R. Copson, chairman) has appointed a task group to start atmospheric tests on new alloys and several older alloys that had not been previously tested. Included are aluminum, magnesium, copper, lead, nickel, molybdenum, tantalum, titanium, zinc, and zirconium metals and alloys. Specimens will be exposed at Kure Beach, N. C. (east coast marine), State College, Pa. (rural), New York Area (industrial), and Point Reyes, Calif. (west coast marine). Specimens 8 by 4 by 0.050 in. will be exposed 2, 7, and up to 20 years. Corrosion will be determined by weight loss, changes in tensile properties, and pit depth.

*Subcommittee VII on Weather* (C. P. Larrabee, chairman) has made calibration tests on the corrosivity of ASTM Atmospheric Test Sites, varying from 1 to 8 years, and will prepare a report on the data to be considered for publication.

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* Presented at the Sixieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 2.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee VIII on Galvanic and Electrolytic Corrosion* (G. V. Kingsley, chairman) is working on the inspection of the final group of stainless steel galvanic couples. The panel type for magnesium couples, Part III, in this test program are expected to be placed on exposure during the summer of 1957.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members; 66 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
*Chairman.*

A. W. TRACY,
*Secretary.*

# REPORT OF COMMITTEE B-4
## ON
# METALLIC MATERIALS FOR ELECTRICAL HEATING, ELECTRICAL RESISTANCE, AND ELECTRICAL CONTACTS*

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance and Electrical Contacts held three meetings during the year: in Swampscott, Mass. on June 13, 1956, in Pittsburgh, Pa. on November 16, 1956, and in Philadelphia, Pa. on March 8, 1957.

The committee consists of 51 members, of whom 22 are classified as producers, 21 as consumers, and 8 as general interest members.

As in 1956, the committee, through Subcommittee IV, sponsored a Symposium on Electrical Contacts held at The Pennsylvania State University the week of June 10, 1957. Many of the speakers on this program are members of Committee B-4.

Upon the recommendation of Subcommittee III on Thermostatic Metals the Society is publishing a compilation of tentatives and standards on thermostat metals. This publication will fill the need for a convenient reference for testing procedures on these materials.

### ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions[1] issued February 15, 1952, of the Standard Method of Accelerated Life Test for Metallic Materials for Electrical Heating (**B 76 – 39**),[1] be approved for reference to letter ballot of the Society, with the note in Section 8 modified to read as follows:

NOTE.—When a glass front is used, the pyrometric filament current shall be adjusted to correct for the absorption and reflection of glass (see Appendix).

### REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated and accordingly asks for the necessary nine tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

**Standard Method of Accelerated Life Test for Metallic Materials for Electrical Heating (B 76 – 39):**[1]

*Appendix.*—In the seventh paragraph, change the fifth sentence to read, "A correction of approximately 10 F (5.5 C) or a correction as determined by a specific test for the conditions involved shall be added to the temperature as observed."

**Standard Specifications for Drawn or Rolled Alloy, 80 Per Cent Nickel, 20 Per Cent Chromium, for Electrical-Heating Elements (B 82 – 52):**[1]

*Section 7.*—Change the first sentence to read, "The nominal resistivity (Note) shall be the resistivity of the wire as quenched from a temperature above 1450 F (760 C)."

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 2.

Change the Note to this section to read as follows:

NOTE.—The characteristics of this alloy are such that the actual resistivity of annealed wire may be as much as 6 per cent higher than nominal depending on its method of manufacture.

*Section 11.*—Change to read as follows:

11. The change in resistance with change in temperature shall not vary beyond the minimum and maximum limits as given in the following table where $\Delta R$ is the actual change in resistance in ohms from the resistance at 77 F (25 C) and $R$ is the resistance in ohms at 77 F (25 C).

| Temperature | $\frac{\Delta R}{R} \times 100$ | | |
|---|---|---|---|
| | Av. | Max. | Min. |
| 2000 F (1093C) | 1.4 | 1.86 | 0.94 |
| 1800 F (982 C) | 0.7 | 1.16 | 0.24 |
| 1600 F (871 C) | 0.3 | 0.76 | −0.16 |
| 1400 F (760 C) | 0.3 | 0.76 | −0.16 |
| 1200 F (649 C) | 0.7 | 1.16 | 0.24 |
| 1000 F (538 C) | 1.9 | 2.36 | 1.44 |
| 900 F (482 C) | 2.3 | 2.76 | 1.84 |
| 800 F (427 C) | 2.2 | 2.66 | 1.74 |
| 600 F (315 C) | 1.7 | 2.16 | 1.24 |
| 400 F (204 C) | 1.1 | 1.56 | 0.64 |
| 200 F (93 C) | 0.4 | 0.86 | 0 |
| 77 F (25 C) | 0 | 0 | 0 |

NOTE.—The electrical properties of this alloy can be changed by heat treatment in the temperature range of 500 to 1450 F (260 to 760 C). The wire as supplied by the manufacturer will normally have been rapidly cooled by quenching from an annealing temperature higher than 1450 F (760 C). It will, therefore, have a resistivity near the minimum for the alloy. After heating or slow cooling in the range 500 to 1450 F (260 to 760 C), the resistivity is increased from a fraction of a per cent to about 6 per cent. The resistivity above 1450 F (760 C) is reproducible regardless of the previous treatment below that temperature.

The values for resistance change with temperature in the above table are based on the room temperature resistance of the sample after cooling at the specified rate as given in the ASTM Standard Method B 70. This resistance normally will be higher than the original resistance of the sample before it was heated. The resistances measured as, and after, the sample cools through the 500 to 1450 F (260 to 760 C) range will depend on the cooling rate. The cooling rate must be carefully controlled if results are to be reproducible.

## Tentative Continued Without Revision

The Tentative Specifications for High-Resistivity, Low-Temperature-Coefficient Wire B 267 – 53 T are being considered for revision and are recommended for continuation as tentative.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be announced at the Annual Meeting.[2]

## Research Activities of Subcommittees

A major part of the activities of all of the subcommittees of Committee B-4 is concerned with research into the physical and chemical effects surrounding the operation of these special materials. Environmental and operating conditions imposed upon these materials are so complicated in many cases that it is necessary to learn what factors must be controlled to provide consistent test results and to find out what is happening to the materials under the test conditions. For these reasons, the main emphasis in this annual report is on the research activities of the various subcommittees. Only the highlights are given and the report does not indicate the amount of time and effort being spent on these problems.

*Subcommittee I on Electrical Heating and Resistance Materials* (C. W. Armstrong, chairman) is developing an accelerated life test for iron-chromium-aluminum alloys for use under high-temperature conditions. Because of the wide range of conditions under which these alloys may be used, the determination of the test specification involves the testing of these materials under widely different conditions. In the past, investigations leading to the determination of test conditions have resulted in the improvement of the alloys under consideration.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee III on Thermostat Metals* (U. U. Savolainen, chairman), in addition to maintaining and revising the specifications which are now being published in pamphlet form, is developing methods for thermal conductivity of thermostatic bimetal, corrosion-resistant properties of bimetals, and emissivity of bimetallic materials.

*Subcommittee IV on Contact Materials* (J. D. Kleis, chairman):

*Section A on Life Tests* (T. K. Knapp, chairman) is working on methods of measuring the oxidation and sulfide resisting properties of electrical contact materials. This investigation involves the oxidation and sulfiding under controlled conditions of various contact materials and then testing the voltage and current characteristics of the contacts. The main problem has been to get consistent data among the various laboratories. Considerable progress has been made to bring some of the variables under control.

*Section B on Physical Properties* (F. R. Farnham, chairman).—While heat conductivity is considered to be a well known property of materials, methods of measurement and procedures to get simple tests are not available. This group has been working on this problem for some time and is beginning to make progress.

*Section C on Standardization of Contact Forms and Sizes* (D. K. Wells, chairman) is preparing sizes and shapes of contacts which will decrease costs to both the consumer and the producer.

*Section D on High Current Contacts* (M. R. Swinehart, chairman) has developed a method for measuring the contact resistance of contacts for high-current applications which has given consistent results among the various laboratories. To investigate the problem of contact wear under heavy current, short circuit conditions, this group has divided the problem into two areas, one of which covers currents up to 600 amp and the other currents in the range over 10,000 amp. These tests will consider erosion, contact resistance, resistance to welding, restriking voltage, contact repulsion, velocity of anode and cathode spots and consider tests under other atmospheres and under vacuum.

*Section E on Bibliography* (E. I. Shobert II, chairman) is keeping up to date the Bibliography on Electrical Contacts (STP 56G) by preparing annual supplements.

*Section F on Microcontacts* (A. L. Van Emden, chairman) is investigating the percentage of operations which could be considered as having made electrical contact under a given set of low-current, low-voltage, and low-pressure conditions. This group has begun to collect data from various laboratories and is making progress on tests under what are called "dry circuit" conditions.

*Section G on Static Connectors and Connections* (C. E. McCarthy, chairman) is starting work on plug-in type connectors and swaged type connections.

A new group on sliding contacts has been organized and will eventually become a section of Subcommittee IV.

In summary, the research activities on electrical contacts are as follows:

1. Study of the electrical properties of films on metallic electrical contact materials.
2. Mechanism of film breakdown and film conduction on electrical contacts.
3. Development of methods for determining electrical properties of contact materials.
4. Methods of evaluating and studying

phenomena occurring at the interfaces of sliding contacts.

5. Methods of evaluating and studying phenomena of stationary or static electrical connections.
6. Methods of evaluating and studying phenomena occurring at the interfaces of contacts carrying currents less than one milliampere (dry circuits).
7. Methods for studying and evaluating phenomena occurring at the interfaces of high-current electrical contacts.
8. Literature surveys.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 42 members returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted in behalf of the committee,

ERLE I. SHOBERT II,
*Chairman.*

C. K. STROBEL,
*Secretary.*

### EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-4 presented to the Society through the Administrative Committee on Standards a new Tentative Recommended Practice for Dimensional Standards for Projection Welding Contacts. The new tentative recommended practice was accepted by the Standards Committee on September 13, 1957, and it appears in the 1957 Supplement to Book of ASTM Standards, Part 2, bearing the designation B 321 – 57 T.

# REPORT OF COMMITTEE B-5

## ON

## COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year: at Hershey, Pa., in October 1956, and at New York, N. Y., in January 1957. At these two sessions, the Executive Subcommittee and Subcommittees W-1, W-2, W-3, W-4, F-1, G-1, and G-3 also met.

The committee regrets to announce the passing of Nicholas H. Murdza, A. A. Baldwin, and A. E. White, faithful and long-time members in committee activities of the Society.

The committee consists of 131 members of whom 129 are voting members; 62 are classified as producers, 49 as consumers, and 18 as general interest.

Considerable study has been given to the Regulations Governing Committee B-5 and several revisions have been adopted.

The American Group for Technical Committee 26 on Copper and Copper Alloys of the International Organization for Standardization held a meeting at Hershey, Pa. Items discussed were classification of coppers, methods of test for copper and copper-alloy tubing (pin test), and method of test for residual (internal) stresses in copper and copper-base-alloy products (mercurous nitrate test).

A new task group on classification of cast-copper alloys (J. S. Roberts, chairman) was organized to handle contacts with the ISO/TC 26 working group on the subject.

Papers were presented at each meeting of Committee B-5, at the October meeting on "Grain Growth and Recrystallization of 2 per cent Beryllium Copper" by George Beatty, formerly of the Beryllium Corp., and at the New York meeting on "Problems Associated with Hardness Conversion of Several Copper Alloys," by J. T. Richards and Ellsworth M. Smith, Penn Precision Co. These papers are appended hereto.[1]

### RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending one specification for publication as tentative, the adoption of three tentatives as standard, and revisions of six standards. These recommendations are covered in detail in Appendix I.[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee W-1 on Plate, Sheet and Strip* (A. E. Moredock, chairman) is considering modifications in Specifications B 122 and B 194.

*Subcommittee W-2 on Rods, Bars and Shapes* (J. D. MacQueen, chairman) recommended that Specification B 133 be adopted as standard.

*Subcommittee W-3 on Wire and Wire Rod* (Sidney Metzger, chairman) recommended retention of Specification B 272 as tentative and revisions of Specification B 159 to eliminate the note in regard to stress relieving Alloy A, spring wire, under Table I.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] See pp. 157 and 161.

[2] See p. 155.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee W-4 on Pipe and Tube* (G. C. Mutch, chairman) prepared the new specification for Copper-Silicon Alloy Seamless Pipe and Tube and revisions of Specifications B 42, B 43, B 75, and B 111 as set forth in the Appendix.

Consideration is being given to revision of hardness requirements in Specifications B 75, B 88, B 135, B 188, B 302 and B 306; revision of chemical requirements for type of tube in Specification B 75; 5S and 10S wall thickness schedules; revision of requirements for expansion test in Specification B 111; statistical sampling for Specification B 75 and other tube specifications; inclusion of alloy D of Specification B 169 in Specification B 111; and revision of wall thickness tolerance requirements in Specifications B 68, B 75, B 135 and B 251.

*Subcommittee F-1 on Castings and Ingots for Remelting* (G. H. Clamer, chairman) has appointed a task group to work with ISO/TC 26 on the classification of cast copper and copper alloys.

Consideration is being given to specification needs for investment castings and copper-nickel additives, revisions of all specifications under its jurisdiction to eliminate discrepancies in form and wording, and coordination of SAE and ASTM standards.

*Subcommittee G-1 on Methods of Test* (G. R. Gohn, chairman) prepared editorial changes in the various product specifications under the jurisdiction of Subcommittees W-1, W-2, W-3, W-4, and F-1 to define more exactly the applicable method of test for the determination of mechanical properties, and, subject to letter ballot approval, it will recommend that the various rod specifications under the jurisdiction of Subcommittee W-2 be revised to clarify the method of tension testing. In the event one party tests a rod specimen in full-size section and the other party tests a machined specimen, the results on the full-size specimen shall be used in case of disagreement to determine conformance to the specification requirements.

The subcommittee is preparing recommended practices for the determination of dimensional tolerances, and a task group is reviewing the latest revision of the Tentative Methods for Determining the Average Grain Size of Metals (E 112), prepared by Committee E-4, to determine whether the revisions completely meet the needs of Committee B-5.

Consideration is being given to revising the Standard Method of Test for Expansion (Pin Test) of Copper and Copper Alloy Tubing (B 153) to agree with ISO recommendations on the angle of the taper pin and to revising the Standard Method of Mercurous Nitrate Test for Copper and Copper Alloy (B 154 – 51) to agree with ISO proposals. Studies are continuing on the relation between thickness of the test strip and Rockwell hardness.

*Subcommittee G-3 on Editorial and Publications* (W. F. Roeser, chairman) is reviewing the specifications under the jurisdiction of Committee B-5 for form, organization, and editorial consistency.

This report has been submitted to letter ballot of the committee, which consists of 131 voting members; 90 members returned their ballots, of whom 81 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
*Chairman.*

L. H. ADAM,
*Secretary.*

## APPENDIX I

# RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1955 Book of ASTM Standards, Part 2, or in the 1956 Supplement to Book of ASTM Standards, Part 2.

### NEW TENTATIVES

The committee recommends for publication as tentative the Specification for Copper-Silicon Alloy Seamless Pipe and Tube, as appended hereto.[1]

### REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

**Standard Specification for Seamless Copper Pipe, Standard Sizes (B 42 – 55):** Revise as follows:

*Section 3.*—Add a new item to read, "(5) If material is required to meet ASME Code (Section 5(*a*))."

*Section 5(a).*—Change to read as follows:

(*a*) All pipe shall normally be furnished in the drawn temper.

NOTE.—Drawn temper pipe specified to meet requirements of the ASME Boiler and Pressure Vessel Code shall have tensile properties as follows:

| Pipe Size Nominal, in. | Tensile Strength, min, psi | Yield Strength,[a] min, psi |
|---|---|---|
| 1/8 to 2, incl. | 45 000 | 40 000 |
| 2½ to 12, incl. | 36 000 | 30 000 |

[a] At 0.5 per cent extension under load.

**Standard Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stocks (B 111 – 56)**

*Section 4.*—In Paragraph (*b*) change "unless otherwise specified" to read "one of which shall be specified." In the Note to this section delete the last sentence.

*Section 16.*—Change Paragraph (*c*) to read as follows:

(*c*) The length of the tubes shall not be less than that specified when measured at a temperature of 20 C (68 F), but may exceed the specified value by the amounts given in the following table:

| Specified Length. ft | Tolerance, all plus, in. |
|---|---|
| Up to 15, incl. | 3/32 |
| Over 15 to 20, incl. | 1/8 |
| Over 20 to 30, incl. | 5/32 |
| Over 30 to 60, incl | 1/4 |
| Over 60 to 100, incl.[a] | 3/8 |

[a] Condenser tubes in lengths over 100 ft are not in present demand. Tolerance values for these lengths will be developed as experience dictates.

Add a new Paragraph (*d*) as follows and reletter the present Paragraph (*d*) as (*e*):

(*d*) *Squareness of Cut.*—The angle of cut of the end of any tube may depart from the square by not more than the following:

---

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 2.

| Tube Outside Diameter, in. | Tolerance, in. |
|---|---|
| Up to 5/8, incl. | 0.010 |
| Over 5/8 | 0.016 per inch of diameter |

*Section 17.*—Change to read as follows:

17. *Workmanship.*—Roundness, straightness, uniformity of the wall thickness, and inner and outer surface of the tube shall be such as to make it suitable for the intended application.

### Standard Specification for Seamless Copper Boiler Tubes (B 13 – 55):

*Section 6.*—Delete the second sentence of the Note.

### Standard Specification for Seamless Red Brass Pipe Standard Sizes (B 43 – 55):

*Section 5.*—Delete the last sentence of the Note.

### Standard Specification for Seamless Copper Tube (B 75 – 55):

*Section 6 (a).*—In the Note, delete "The tension test shall be required only when definitely specified in the purchase order."

### Standard Specification for Phosphor Bronze Wire (B 159 – 54):

*Section 1.*—Delete the Note.

## ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

*Tentative Specification for:*
Copper Rod, Bar, and Shapes (**B 133 – 54 T**),
Threadless Copper Pipe (**B 302 – 56 T**), and
Copper Drainage Tube (DWV) (**B 306 – 56 T**).

## APPENDIX II

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# THE EFFECT OF ANNEALING TIME, TEMPERATURE, AND PRIOR COLD WORK UPON RECRYSTALLIZATION AND GRAIN GROWTH OF 2 PER CENT BERYLLIUM COPPER*

By George R. Beatty[1]

The material used in this work was the commercially standard 2 per cent beryllium copper alloy with the following analysis:

| | |
|---|---|
| Beryllium, per cent | 1.96 |
| Iron, per cent | 0.11 |
| Silicon, per cent | 0.15 |
| Aluminum, per cent | 0.05 |
| Cobalt, per cent | 0.26 |
| Tin, per cent | 0.01 |
| Lead, per cent | 0.00 |
| Zinc, per cent | 0.03 |
| Nickel, per cent | 0.02 |
| Chromium, per cent | 0.01 |
| Manganese, per cent | 0.01 |
| Silver, per cent | 0.01 |

Material was obtained from $\frac{1}{4}$ in. hot-rolled stock that had been slab milled. This material was rolled to 0.100 in. (60 per cent reduction) and annealed. The resulting grain size was 0.050 mm. Following this, portions of this stock coil were rolled from 10 per cent to 90 per cent in increments of 10 per cent. At finish gage, this material was slit to $\frac{1}{2}$ in., and 2 in. long samples were cut. These 2-in. samples were used for the annealing studies. The following temperatures were under consideration: 1400 F, 1450 F, and 1500 F. These temperatures were chosen since they represent the solution-annealing range for 2 per cent beryllium copper. In addition to the variable of cold work and temperature, a further variable of time was tested. This time ranged from one second up to 8 hr. The annealing of 45 min or over was done in a resistance air circulating furnace, with thermocouples placed on top of the samples. Between 1 sec and 30 min the annealing was done in a salt bath with an immersion thermocouple connected with the temperature controller. In each case, the samples were quenched in running water immediately following the anneal.

### Effect of Time at Constant Temperature

The purpose of the first part of this study was to determine the effect of time with the temperature remaining constant at each of the three temperature levels (Fig. 1).

*Results at 1400 F:*

It was found that recrystallization started at approximately 15 min and was completed at the end of approximately 30 min. It was also noticed that there was a difference in recrystallized grain size between the 10 per cent and the 90 per cent cold-rolled material, with the 10 per cent material having a grain size of approximately 0.045 mm.

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* Presented at a Meeting of Committee B-5 held at Hershey, Pa., October 1956.

[1] Capitol Products Corp., Mechanicsburg, Pa. (Formerly Beryllium Corp., Reading, Pa.)

while the 90 per cent cold-rolled material had a grain size of 0.020 mm.

It will be noticed in the 1400 F curve on Fig. 1 (60 per cent hard material) that between 15 and 45 min rapid grain growth occurs, up to a grain size of approximately 0.030 mm. After this point, the rate of grain growth changes appreciably, and at the end of 8 hr a grain size of 0.045 mm is reached.

*Results at 1450 F:*

The second curve on Fig. 1 illustrates the effect of temperature at 1450 F upon grain growth for 60 per cent hard material. It can be noted that the start of recrystallization occurs in approximately 45 sec, and the material is fully recrystallized at the end of 3 min. Again a large difference was noted between the initially recrystallized grains for 10 per cent hard material and 90 per cent hard material, with the 10 per cent material having an initial grain size of 0.035 mm while the 90 per cent hard material had an initial grain size of 0.015 mm.

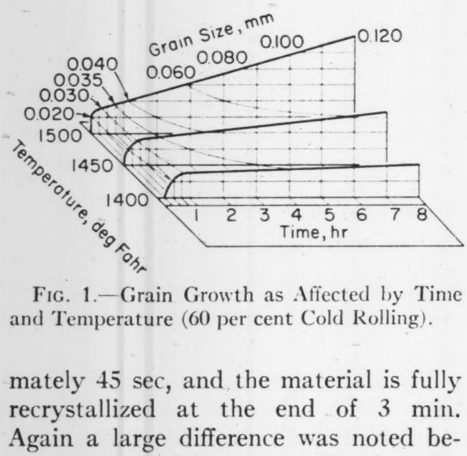

FIG. 1.—Grain Growth as Affected by Time and Temperature (60 per cent Cold Rolling).

In Fig. 1 (1450 F) it will be noticed that grain growth is very rapid up to approximately 30 min, with a grain size of 0.030 mm. From this time until 8 hr, the rate decreases and the resultant grain size at 8 hr is 0.065 mm.

*Results at 1500 F:*

The third curve on Fig. 1 shows recrystallization occurring in less than 45 sec, with the material being fully recrystallized at the end of 1 min. Again it was noted that the 10 per cent hard material had an initial grain size of 0.035 mm, while the initially recrystallized 90 per cent hard material had a grain size of 0.015 mm. It can be seen on this curve for the 60 per cent hard material at 1500 F that grains grow rapidly up to approximately 3 min and reach a grain size at this point of 0.025 mm. From this time until 8 hr, the rate is fairly constant (but higher than at 1400 F or 1450 F), with a grain size of 0.120 mm being reached at the end of 8 hr.

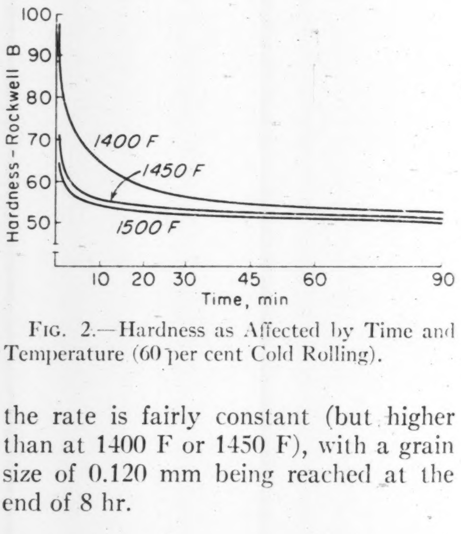

FIG. 2.—Hardness as Affected by Time and Temperature (60 per cent Cold Rolling).

*Comments:*

1. In general, the following conclusion can be drawn from this portion of the work. Lower temperatures (1400 F to 1450 F) will result in uniform grain growth up to 8 hr, whereas the high temperature (1500 F) results in rapid and erratic grain growth, with evidence of duplex grains at the far end of the curve.

2. It would also appear that the re-

crystallized grain size is more dependent upon prior reduction than time or temperature of annealing.

3. It was also noted that the combination of high temperature and low reduction produced erratic and duplexed grains (greater than 0.200 mm).

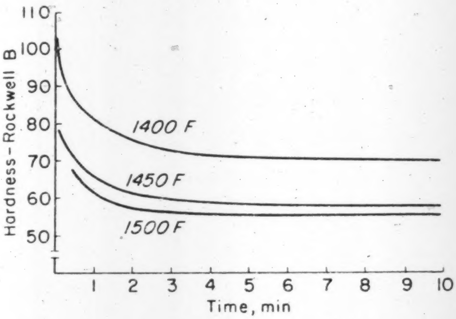

FIG. 3.—Hardness as Affected by Time and Temperature (60 per cent Cold Rolling).

An example of this is shown in the specimen annealed at 1500 F:

| Time | Grain Size | Per Cent Reduction | Change in Grain Size | 8 hr. Grain Size, mm |
|---|---|---|---|---|
| 1.... | 0.065 | 10 | 0.065 to 0.200+ | 0.200+ |
| 2.... | 0.045 | 80 | 0.035 to 0.200+ | 0.200+ (one grain across) |
| 4.... | 0.120 | 20 | 0.035 to 0.200+ | 0.200+ |
| 5.... | 0.090 | 30 | 0.035 to 0.150 | 0.150 (uniform) |
| 5.... | 0.090 | 40 | 0.035 to 0.150 | 0.140 (uniform) |
| 6.... | 0.090 | 50 | 0.035 to 0.150 | 0.130 (uniform) |

4. It is obvious from the curves in Fig. 1 that the time for recrystallization decreased with increasing temperature. It should also be noted that the prior cold work appeared to have no significant effect upon recrystallization time. This is apparently due to the fact that the high temperatures used were such as to overshadow the effect of prior reduction. However, there was some indication that the classical rule "less time for recrystallization with greater cold work" would apply.

## EFFECT OF COLD WORK

It was found that the greater the amount of cold work, the smaller was the initially recrystallized grain; and in like manner, the resultant grain size at the end of 8 hr was smaller with greater amounts of cold work. An example of this is as follows:

For the 1450 F Anneal:

| Per Cent Reduction | Grain Size, mm |
|---|---|
| 10 to 20........ | 0.030 to 0.035 initial<br>0.065 final (8 hr) |
| 30 to 70........ | 0.020 to 0.025 initial<br>0.065 final (8 hr) |
| 80 to 90........ | 0.010 to 0.015 initial<br>0.040 final (8 hr) |

At this time, it should be again mentioned that this work was done on material with an initial grain size of 0.050 mm prior to cold rolling. Undoubtedly, a larger or smaller grain size would materially change the results mentioned in this paper. It is planned to investigate this in the future.

## RECRYSTALLIZATION

Since this study was made with relatively high temperatures, it was not possible to define with any great degree of accuracy the actual beginning and end of recrystallization. However, by plotting hardness values (Rockwell B) *versus* time at 1400 F, 1450 F, and 1500 F it was possible to correlate (in a rough manner) the point of initial softening with microstructure.

Figure 2 shows the drop in hardness at the 3 temperatures under consideration *versus* time up to 90 min for 60 per cent hard material.

Figure 3 is the same as Fig. 2 except that it shows the part of the curve up to 10 min.

Taking into account the curves shown on Fig. 3 and microstructure, it appears that recrystallization begins and ends as follows:

| Temperature, deg Fahr | Start | End | Rockwell Hardness, B Scale |
|---|---|---|---|
| 1400..... | 15 min | 25 to 30 min | 58 |
| 1450..... | 45 sec | 3 min | 59 |
| 1500..... | 45 sec | 1 min | 59 |

Initial hardnesses on the cold-rolled strip range from Rockwell hardness B 90 on the 10 per cent hard material to approximately Rockwell hardness B 107 to 109 for the 80 per cent hard material. In the cases presented on Figs. 2 and 3, this 60 per cent hard material had a cold rolled hardness of Rockwell hardness B 104. Indications are that fully recrystallized 2 per cent beryllium copper has a Rockwell B hardness of <60.

## SUMMARY

In conclusion, there are several statements concerning grain growth and recrystallization of 2 per cent beryllium copper that can be derived from general knowledge concerning the mechanisms of this phenomenon and what has been found out in this work.

1. Grain growth is in general affected by time and temperature, composition, and prior deformation.

A. *Time and Temperature.*—From the work performed in connection with this paper, it appears that the two lower temperatures (1400 F, 1450 F) will result in a more uniform grain growth than in 1500 F temperature.

B. *Composition.*—Although no work has been done along this line, it is fairly obvious that composition would have an effect upon grain growth and recrystallization.

C. *Prior Deformation.*—It was found that with increasing deformation, and all other factors remaining constant, a smaller initial grain size and final grain size can be expected.

2. The time-honored laws of recrystallization state that the time for the beginning or end of recrystallization is less with higher temperature or greater amounts of deformation. The evidence found in this work is in accordance with the first part of this statement. However, the effect of deformation was not observed with any great degree of accuracy. It is felt that the variable of deformation was overshadowed by the variable of temperature, since this work was all performed at a relatively high temperature.

3. It was also noted that a combination of light reduction and high annealing temperature produces erratic and duplex grains over 0.200 mm.

*Acknowledgments:*

The author wishes to thank Mr. M. J. Donachie for his constructive criticism during the preparation of this paper and Mr. F. Jarsocrak for his aid in the laboratory. In addition, a word of thanks to Firth Sterling, Inc. for the use of their salt baths for the short-time anneals and The Beryllium Corp. for sponsoring and supporting this work and their permission to present this paper.

## APPENDIX III

# PROBLEMS ASSOCIATED WITH HARDNESS CONVERSION OF SEVERAL COPPER ALLOYS*

By John T. Richards[1] and Ellsworth M. Smith[1]

## Synopsis

Superficial hardness and tension tests have been conducted upon beryllium copper and phosphor bronze strip to determine hardness conversion data. The effects of strip thickness, temper, and heat treatment were examined and found to influence conversion data materially. For strip thicknesses under 0.030 in., superficial hardness tests may prove unreliable.

On the basis of the information presented, existing specification limits may warrant revision, particularly those applicable to beryllium copper.

Hardness testing has become a standard inspection procedure because of its simplicity. Compared with other inspection techniques, the results are rapidly obtained and are relatively inexpensive. At the same time, the equipment is compact and the initial cost relatively low.

Hardness measurements are frequently employed as an acceptance test for strip materials as well as for many components blanked and formed from strip. In fact, hardness may be the only practical test for many small components, since size and shape may preclude tension testing. This condition is especially true in considering beryllium copper and phosphor bronze, where many small, but vital, parts are fabricated from thin strip.

The hardness values obtained have little direct value but serve as an indirect measure of tensile characteristics. From design or performance standpoints, hardness values are mainly useful to identify a certain level of tensile or yield strength, or perhaps proportional limit.

Several important factors affect the relationship between tensile properties and hardness:

1. Ability of hardness values to be reproduced.
2. Fundamental differences between hardness tests and tension tests.
3. Certain inherent problems associated with hardness testing.

Hardness tests are reproducible provided the equipment is kept in normal repair and proper care is exercised by operating personnel. Difficulties of interpretation may result since the hardness test is local in nature, whereas the tension test covers a larger area and gives an average value for the many local areas contained in the test length. Other factors which may influence the result are possible hardness gradients across the thickness, preferred orientation, support of the specimen, etc.

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* Presented at a Meeting of Committee B-5 held in New York, January, 1957.

[1] Chief Engineer, and Chief Metallurgist, respectively, Penn Precision Products, Inc., Reading, Pa.

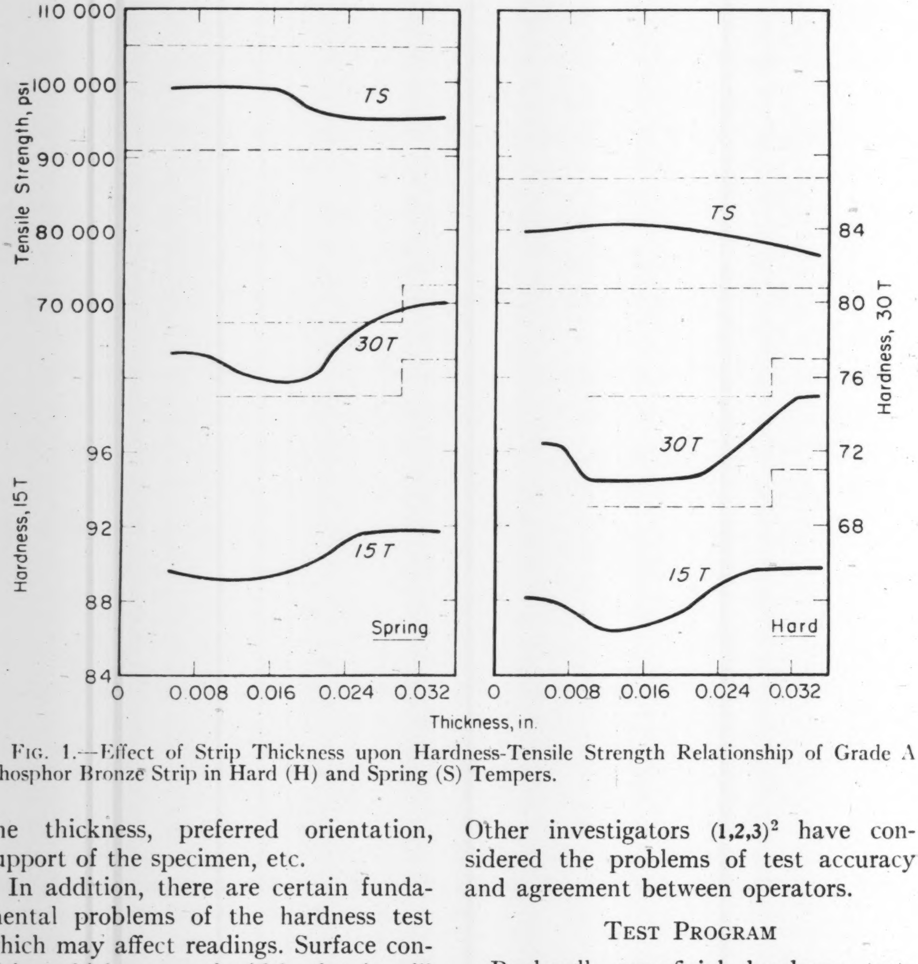

FIG. 1.—Effect of Strip Thickness upon Hardness-Tensile Strength Relationship of Grade A Phosphor Bronze Strip in Hard (H) and Spring (S) Tempers.

In addition, there are certain fundamental problems of the hardness test which may affect readings. Surface condition, thickness, and width of strip will influence results. Type of material being tested may also introduce other variables. It is known, for example, that hardness conversion is dependent upon microstructure which, in turn, is affected by the degree of cold work as well as age-hardening time and temperature in precipitation-hardening materials.

In the present investigation problems of hardness test accuracy and ability to be reproduced are not considered, since it must first be determined whether there exists a definite correlation between tensile values and hardness test results. Other investigators (**1,2,3**)[2] have considered the problems of test accuracy and agreement between operators.

## TEST PROGRAM

Rockwell superficial hardness tests were employed, since they are used extensively for thin strip. Hardness of annealed and rolled tempers was measured on 15T and 30T scales, while 15N and 30N scales were best suited for heat-treated conditions. Diamond pyramid hardness values were also obtained with a Vickers tester using a 2½-kg load. Standard test procedures were employed throughout for tension and hardness, and reported values are averages for two to five tests.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 169.

The material studied included the standard 2 per cent beryllium copper alloy meeting the requirements of ASTM Specification B 194[3] and grade A phosphor bronze (ASTM Specification B 103).[4] A number of lots were employed, some specially prepared and others from stock.

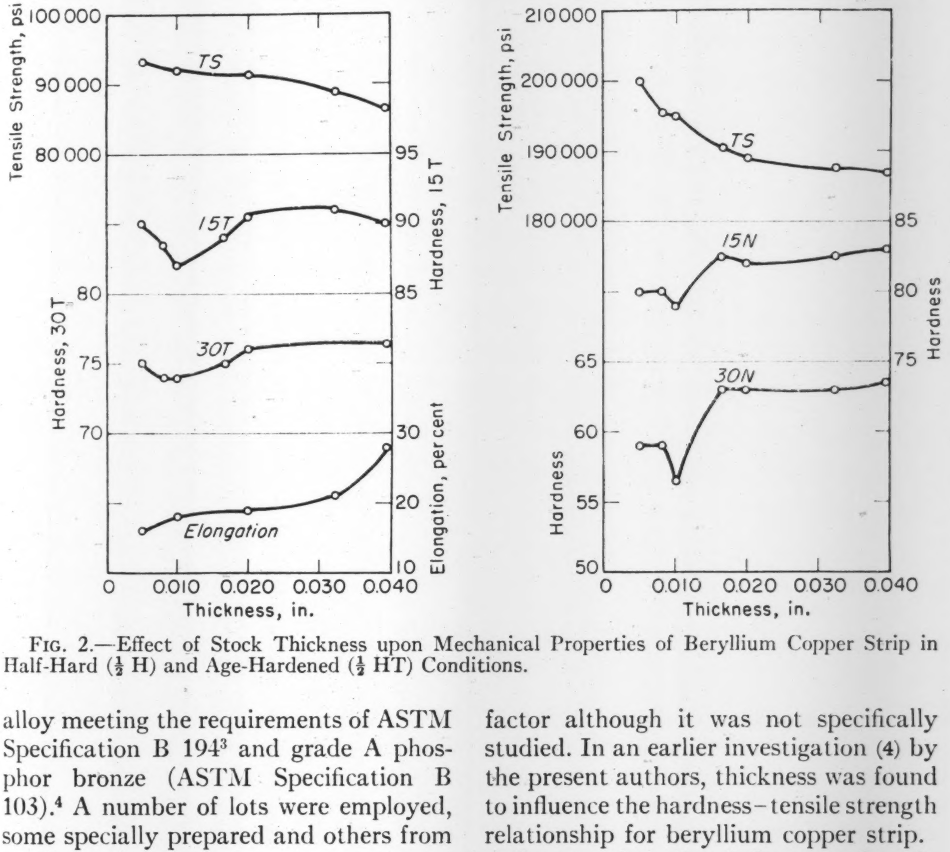

FIG. 2.—Effect of Stock Thickness upon Mechanical Properties of Beryllium Copper Strip in Half-Hard (½ H) and Age-Hardened (½ HT) Conditions.

## TEST DATA

In order to determine the hardness conversion relationships, the effects of thickness, temper, and heat treatment have been investigated.

### *Effect of Thickness:*

Thickness of the test specimen plays a major role in the hardness values obtained. In results presented by others (**1,2,3**), thickness was shown to be a factor although it was not specifically studied. In an earlier investigation (**4**) by the present authors, thickness was found to influence the hardness–tensile strength relationship for beryllium copper strip.

To examine the influence of strip thickness upon hardness, a number of lots of phosphor bronze strip 0.003 to 0.035 in. thick were taken from stock for tension and superficial hardness tests. Lots were tested in either hard (4 numbers hard) or spring (8 numbers hard) temper. The results are shown in Fig. 1.

In this figure, broken lines indicate specification limits for tensile strength and 30T hardness. There is an increase in the hardness range at 0.030 in. in accordance with Specification B 103 for phosphor bronze strip. Solid curves represent average results for several hundred tests. Tensile strength values vary from these curves to the extent of ±2500 psi, while 30T hardness readings run ±1 point and 15T values ±$\frac{1}{2}$ point. In spite of an increase in tensile strength with decreasing thickness, there is a definite drop in hardness for sizes below 0.032 in., followed by a slight rise at 0.012 in. thick.

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[3] Specifications for Copper-Beryllium Alloy Plate, Sheet, Strip, and Rolled Bar (B 194 – 55), 1955 Book of ASTM Standards, Part 2, p. 330.

[4] Specifications for Phosphor Bronze Plate, Sheet, Strip, and Rolled Bar (B 103 – 55), 1955 Book of ASTM Standards, Part 2, p. 160.

Similar tests were conducted on beryllium copper strip; however, for these tests material originating from one coil was employed to eliminate possible variations in composition (4). Figure 2 shows the effect of strip thickness upon mechanical properties of half-hard ($\frac{1}{2}$H) and age-hardened ($\frac{1}{2}$HT) conditions. In both cases, tensile strength increases with a decrease in thickness. The pattern of all four hardness curves is similar in that a sudden drop occurs at thicknesses below 0.020 in. followed by a subsequent rise under 0.010 in. This increase at light gages is probably due to the hardness of the test machine anvil.

In Fig. 3, the influence is shown of stock thickness upon the 15T and 30T hardness of the four standard tempers of beryllium copper prior to heat treatment. For this series of tests, a single coil was again employed to eliminate variations due to composition, while processing was carefully controlled to provide uniform rolling schedules. Solution-annealing times were selected to produce grain sizes ranging from 0.015 to 0.020 mm.

The hardness curves of Fig. 3 reveal an interesting trend because the hardness drop not only becomes more pronounced with softer tempers, but the drop occurs at heavier thicknesses. This is not surprising, since deeper penetration would be expected with softer tempers. By comparing the two sides of Fig. 3, it is

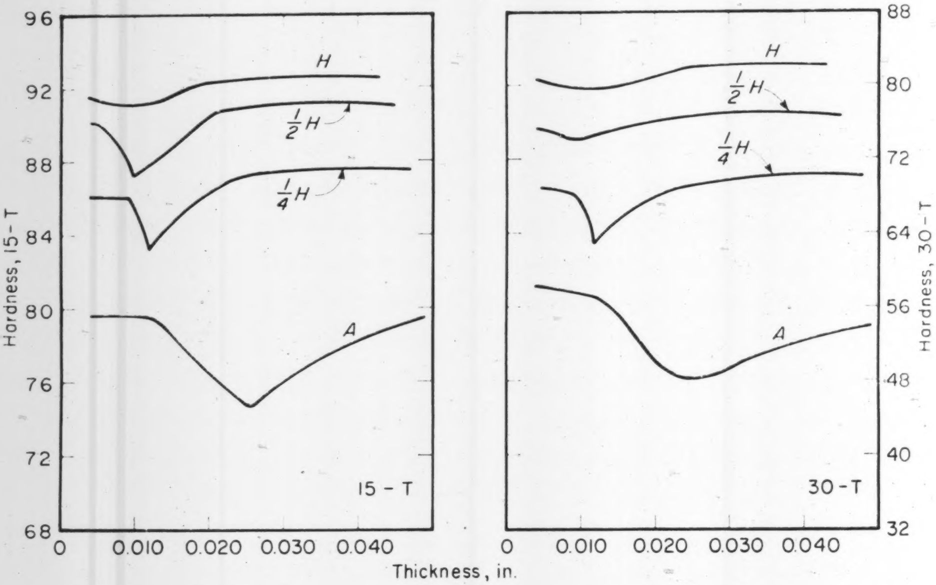

FIG. 3.—Effect of Temper and Thickness upon Superficial Hardness of Beryllium Copper Strip.

apparent that the effect of thickness is similar for 15T and 30T tests. Hardness increments on the right-hand or 30T scale are double those of the left side (15T scale), so that the drop in hardness is twice as great for 30T tests. This is to be expected, because a greater load would cause deeper penetration.

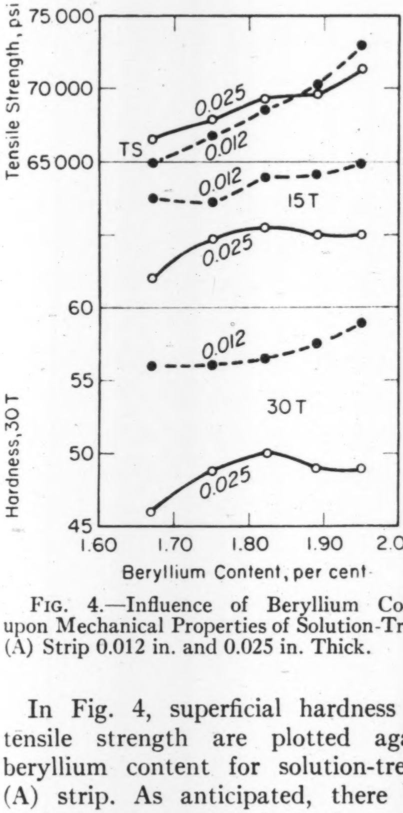

FIG. 4.—Influence of Beryllium Content upon Mechanical Properties of Solution-Treated (A) Strip 0.012 in. and 0.025 in. Thick.

In Fig. 4, superficial hardness and tensile strength are plotted against beryllium content for solution-treated (A) strip. As anticipated, there is a gradual improvement in both strength and hardness with increasing beryllium content. Size effects between 0.012 and 0.025 in. thick strip are negligible for tensile strength but are considerable for hardness. The substantially higher hardnesses shown for strip 0.010 in. thick could be predicted from Fig. 3, since the low point in the curves for solution-treated (A) strip occurs at 0.025 in. Incidentally, beryllium content was not found to be a factor in the conversion relationship for the range studied of 1.67 to 1.95 per cent beryllium.

Figure 5, which indicates the relationship between mechanical properties and aging time at 700 F for half-hard ($\frac{1}{2}$H) strip, reveals a trend similar to that shown in Fig. 2. In spite of higher tensile strength, thinner strip (0.010 in.) has a much lower hardness than heavier material (0.020 in.). Once again it is apparent that anvil hardness is not a factor for half-hard strip at 0.010 in. but may affect the hardness of thinner or softer material. Lack of sensitivity of the hardness test is also demonstrated in that the increase in tensile strength from 190,000 psi to 203,000 psi between 10 and 30 min is not accompanied by an increase in hardness.

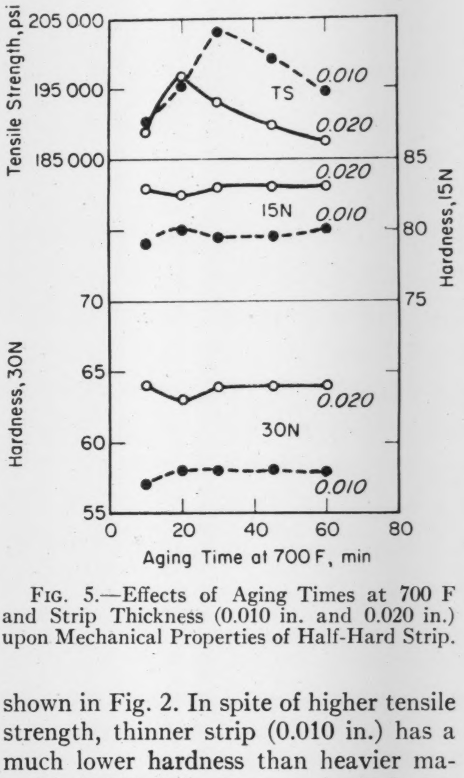

FIG. 5.—Effects of Aging Times at 700 F and Strip Thickness (0.010 in. and 0.020 in.) upon Mechanical Properties of Half-Hard Strip.

All of the tests conducted, including several alloys and tempers, indicate an increase in tensile strength with a decrease in thickness (5). On the other hand, hardness invariably drops, with the magnitude and location of the decline depending upon alloy and temper.

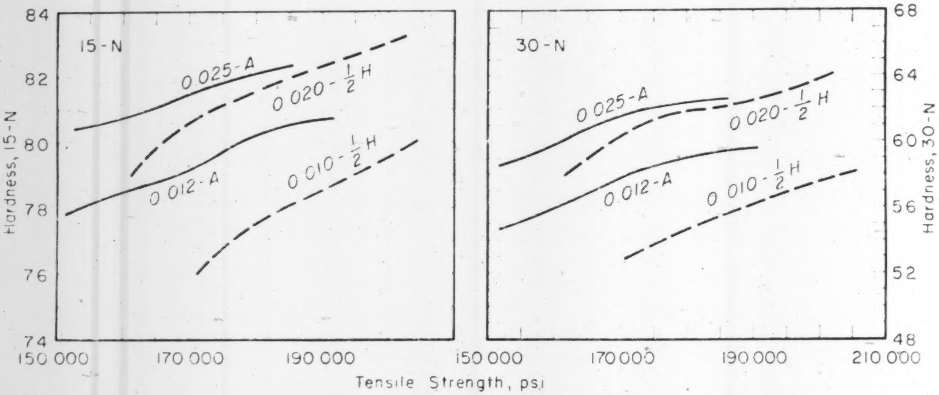

FIG. 6.—Superficial Hardness-Tensile Strength Relationship for Beryllium Copper Strip Age-Hardened From the Solution-Treated (0.012 and 0.025 in. thick) and Half-Hard (0.010 and 0.020 in. thick) Conditions.

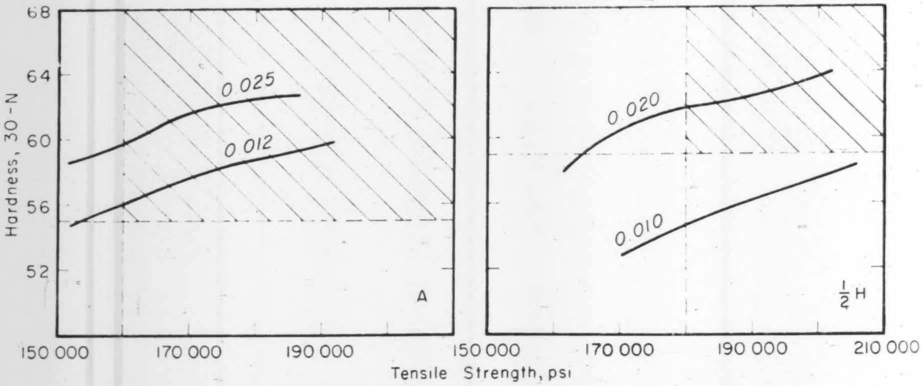

FIG. 7.—Effect of Strip Thickness upon Hardness-Tensile Strength Relationship for Precipitation-Hardened Beryllium Copper. Specification minimums are indicated by broken lines, while cross-hatched area meets minimum requirements for both hardness and tensile strength.

*Effect of Temper:*

To determine the effect of temper upon the conversion relationship for beryllium copper strip, specimens 0.010 to 0.025 in. thick were heat treated for various times from 5 min up to the time required for peak hardness. The results for solution-treated (A) and half-hard (½H) material are given in Fig. 6.

In addition to the influence of thickness, it is apparent that different conversion relationships exist for the two tempers tested. For a given tensile strength, the solution-treated condition produces higher hardness than rolled material of the half-hard temper.

Figure 7 presents the 30N data given in Fig. 6 but in slightly different form. The broken lines indicate minimum tensile strength and hardness limits in accordance with ASTM Specification B 194. The cross-hatched area includes points which meet both specification minimums. It is apparent that it is easier to comply with hardness minimums than tensile strength limits. This is especially true for solution-treated (A) material. The right-hand side of Fig. 7 clearly illustrates the effect of thickness upon the hardness conversion relationship, since 0.010 in. thick material in most instances meets the minimum tensile requirement of 180,000 psi, but none of the points attains the minimum hardness limit of 30N 59.

TABLE I.—EFFECT OF DIRECTIONALITY UPON HARDNESS-TENSILE STRENGTH RELATIONSHIP.

| Alloy | Type | Temper | Thickness, in. | Superficial Hardness | | Tensile Strength, psi | | |
|---|---|---|---|---|---|---|---|---|
| | | | | 15T | 30T | 0 deg | 45 deg | 90 deg |
| Beryllium Copper..... | 25 | H | 0.020 | 93.0 | 82.5 | 115 000 | 117 000 | 123 500 |
| Phosphor Bronze...... | Grade C | S | 0.020 | 93.0 | 81.0 | 115 500 | 116 800 | 124 000 |
| Phosphor Bronze...... | Grade A | XS | 0.010 | 90.5 | 79.5 | 106 500 | 111 000 | 117 500 |
| Brass................ | 70-30 | XS | 0.010 | 89.5 | 78.0 | 109 800 | 113 500 | 121 800 |

As copper alloys are rolled, they develop a directional pattern with respect to tensile properties through preferred orientation (6). From a performance standpoint, directionality may influence the conversion relationship. This condition is shown in Table I. Tensile strength is shown to vary rather substantially with the angle of the rolling direction, although only one hardness value can be determined. Consequently, if the hardness is indicative of tensile strength in the longitudinal direction, it might understate transverse strength where directionality is present.

In addition, the degree of directionality may vary along the thickness of the strip. For example, one directional pattern may be found directly below the surface at the point of the hardness reading, but this condition may not apply throughout the cross-section. Light rolling reductions, such as a planish pass, may only harden the surface of the strip, leaving the center soft. Because the hardness test will measure hardness near the surface, this reading may not correspond with the tensile test, which yields an average value for the entire cross-section.

*Effect of Heat Treatment:*

Since beryllium copper responds to precipitation hardening, varying degrees of heat treatment will produce different structures and, conceivably, could influence the hardness conversion relationship. To test this possibility, a number of underaging and overaging heat treatments were applied to produce various levels of strength and hardness.

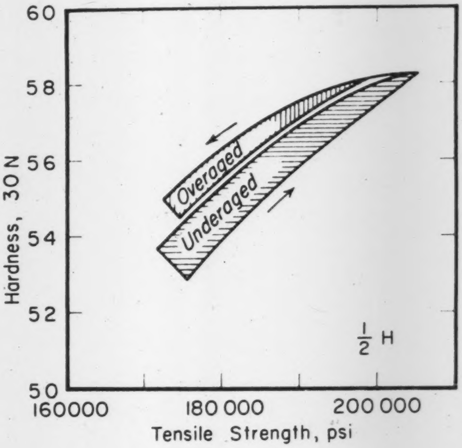

FIG. 8.—Influence of Aged Condition upon Hardness-Tensile Relationship for Half-Hard Beryllium Copper. Approximately 95 per cent of all points fall within scatter bands.

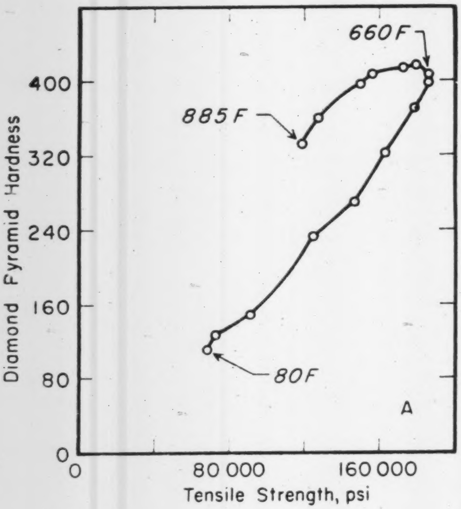

FIG. 9.—Vickers Hardness-Tensile Strength Relationship for Solution-treated (A) Beryllium Copper as Affected by Aging for One Hour at Temperatures Ranging from 80 to 885 F.

companies overaging. It has been shown (8) that the Vickers hardness of a grain may vary from 390 in the center to 260 at the boundary. This softness at the boundary is also an indication of low strength, so that the greater sensitivity of a tension test will reflect this weakness, whereas the averaging characteristic of the hardness test will probably be largely influenced by the hardness of the grain center.

Further tests were conducted to verify this condition. In Fig. 9, specimens were heated for 1 hr at various aging temperatures, and it is apparent that substantially higher hardnesses are found in the overaged condition. In the case of material having a tensile strength of 120,000 psi in the underaged condition, a Vickers hardness of 228 is obtained in comparison with a hardness of 332 for strip overaged to the same tensile level. From an inspection standpoint, variations of this degree would be completely misleading.

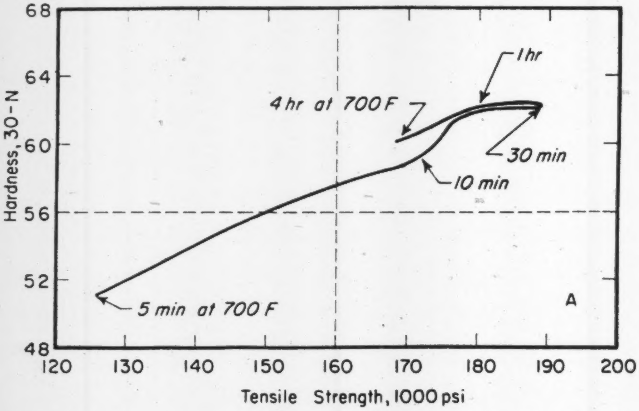

FIG. 10.—Influence of Aging Time at 700 F upon Hardness-Tensile Strength Relationship of Solution-treated (A) Beryllium Copper. Specification minimums are indicated by broken lines.

The results are shown in Fig. 8. The scatter bands encompass approximately 95 per cent of plotted points. In all curves of this type, it was found that overaged material has substantially higher hardness than underaged material of the same tensile strength (7). This condition may result from the presence of excessive grain boundary precipitate which ac-

In Fig. 10, aging time has been varied while temperature was held constant at 700 F. Similarly, overaged material has a slightly higher hardness than underaged material of the same tensile level.

This effect, however, is not as pronounced as in Fig. 9, since the degree of overaging was not as great.

## CONCLUSIONS

The following conclusions can be drawn:

1. The usefulness of superficial hardness tests for phosphor bronze strip thinner than 0.030 in. is questionable, unless thickness-induced limitations are recognized.

2. Superficial hardness tests for beryllium copper under 0.016 in. thick for harder tempers or 0.030 in. for the solution-treated condition may prove misleading.

3. Although these tests reveal a drop in hardness with a decrease in thickness, a corresponding decline in tensile strength, yield strength, or proportional limit was not observed. Unfortunately, this failing of the hardness test presents a serious problem, since the decreases noted occur at varying points for different materials and tempers.

4. Fairly good agreement is shown to exist between the superficial 15N and 30N scales as well as the 15T and 30T scales.

5. Structural problems combined with certain thickness limitations probably preclude the development of useful hardness conversion data for beryllium copper.

6. In addition, the hardness limits of beryllium copper appear too low with respect to the tensile minimums now in effect for ASTM Specification B 194. This condition is evident in Fig. 7. Since there is no difficulty in meeting the tensile strength minimums, it probably would be desirable to increase hardness limits.

In order to study some of the anomalous conditions uncovered, further tests are being conducted and will be reported at a later date. Other limitations of hardness tests are being investigated, while apparent discrepancies in current hardness specification limits are also being reviewed in the hope of producing more realistic values.

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## REFERENCES

(1) H. N. Van Deusen, L. I. Shaw, and C. H. Davis, "Physical Properties and Methods of Test for Sheet Brass," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, pp. 173–210 (1927).

(2) J. R. Townsend, W. A. Straw, and C. H. Davis, "Physical Properties and Methods of Test for Some Sheet Non-Ferrous Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, pp. 321–356 (1929).

(3) G. R. Gohn, "A Hardness Conversion Table for Copper-Beryllium Alloy Strip," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, pp. 230–239, (1955).

(4) J. T. Richards and E. M. Smith, "The Properties of Beryllium Copper Strip as Affected by Cold Rolling and Heat Treatment," 1956. West Coast Papers on Metals (Issued as separate publication *ASTM STP No. 196*).

(5) J. T. Richards, "How to Get More Out of Beryllium Copper," *Materials and Methods*, Vol. 44, No. 6, pp. 112–114, (Dec. 1956).

(6) J. T. Richards and E. M. Smith, "Factors Affecting the Forming Properties of Several Copper Alloys in Strip Form," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 867 (1956).

(7) J. T. Richards, "New Heat Treatments Improve Beryllium Copper," *Iron Age*, Vol. 178, No. 19, pp. 95–98 Nov. 8, 1956.

(8) J. T. Richards, "A Review of Beryllium and Beryllium Alloys," *Journal of Metals*, Vol. 3, pp. 379–386, May, 1951.

# REPORT OF COMMITTEE B-6
## ON
# DIE-CAST METALS AND ALLOYS*

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: in Atlantic City, N. J., on June 20, 1956, and in Philadelphia, Pa., on February 6, 1957.

The committee consists of 92 members, of whom 44 are classified as producers, 21 as consumers and 27 as general-interest members.

## Revision of Tentatives

The committee recommends revisions as indicated of the following three tentative specifications and continuation of the specifications as tentative:

### Tentative Specification for Aluminum-Base Alloy Die Castings (B 85 – 56 T):[1]

*Section 1.*—Change "Eleven" to read "Nine."

*New Section.*—Add the following new Section 2 entitled "Basis of Purchase," and renumber the subsequent sections accordingly:

> 2. Orders for die castings under these specifitions shall include the following information:
> (*1*) Quantity: (number of pieces of each die casting),
> (*2*) Alloy (see Table I),
> (*3*) Drawing of die casting giving all necessary dimensions and showing amount of finish left for machining, if any; location of ejector pin marks or parting lines shall be at option of manufacturer, unless specifically designated on the drawing, and
> (*4*) Any special inspection requirements in addition to the regular requirements listed in this specification shall be mutually agreed upon by the manufacturer and the purchaser.

*Section 4.*—Change to read as follows:

> 4. The chemical analysis shall be made in accordance with the Standard Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys (ASTM Designation: E 34), or by any other approved method agreed upon by the manufacturer and the purchaser. The analysis may be made spectrographically, provided that in the case of dispute, the results secured by Methods E 34 shall be the basis for acceptance.

*Section 9.*—In the heading and in line 5, change "physical properties" to read "mechanical properties."

*Table I.*—Delete alloys SC54A and SC54B, and delete reference to these alloys in Footnote (*c*).

*Table II.*—Delete alloys SC54A and SC54B. Under "Die-Filling Capacity" for alloy G8A change "4" to read "5," and under "Resistance to Corrosion" for Alloys SC84A, SC84B and SC114A change "4" to read "5."

### Tentative Specification for Zinc-Base Alloy Die Castings (B 86 – 53 T):[2]

*Section 1.*—Delete reference to alloy AC43A.

*Footnote 3.*—Change to read, "SAE Specifications No. 903 and 925 conform to the requirements for alloys AG40A and AC41A, respectively."

*New Section.*—Add a new Section 2 on "Basis of Purchase" as recommended above for Specifications B 85, and renumber the subsequent sections accordingly.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1956 Supplement to Book of ASTM Standards. Part 2.

[2] 1955 Book of ASTM Standards, Part 2.

*Table I.*—Delete alloy AC43A. Change the maximum copper content in Alloy AG40A from "0.15" to read "0.25" per cent. Add the following new Footnotes (*c*) referencing after the title of Table I, and new footnote (*d*) referencing after the copper content for alloy AG40A:

(*c*) Zinc alloy die castings may contain nickel, chromium, silicon, and manganese in amounts up to their solubility (0.02, 0.02, 0.035, and about 0.5 per cent, respectively) at the freezing temperature. No harmful effects have ever been noted due to the presence of these elements in these concentrations and, therefore, analyses are not required for these elements.

(*d*) For the majority of commercial applications, a copper content in the range of 0.25 to 0.75 per cent will not adversely affect the serviceability of die castings and should not serve as a basis for rejection.

*Section 8.*—Retitle "Mechanical Properties and Tests" and replace with the following:

8. Unless specified in the order or specifically guaranteed by the manufacturer, acceptance of die castings under these specifications shall not depend on mechanical properties determined by tension or impact tests. When tension or impact tests are made, the test specimens and procedures shall be as shown in the Appendix to these specifications.

*Appendix.*—Change the table in the appendix to read as follows:

| Alloy | Tension Tests on Round Specimens | | Charpy Impact on Square Specimens, ft-lb | Brinell Hardness Number on Square Specimens, 500-kg Load, 10-mm Ball, 30 sec. |
|---|---|---|---|---|
| | Tensile Strength, psi | Elongation in 2 in., per cent | | |
| AG40A (XXIII)... | 41 000 | 10 | 43 | 82 |
| AC41A[a] (XXV).... | 47 000 | 7 | 48 | 91 |

[a] Die castings of alloy AC41A shall not be used in applications where they will be subjected to prolonged temperatures above 200 F.

**Tentative Specification for Zinc-Base Alloys in Ingot Form for Die Castings (B 240 - 52 T):**[2]

*Section 1.*—In the second sentence, change "Three" to read "Two."

*Table I.*—Delete alloy AC43A. Add the following footnote referenced after the table title:

Zinc-base alloy ingot for die casting may contain nickel, chromium, silicon, and manganese in amounts up to their solubility (0.02, 0.02, 0.035, and about 0.5 per cent respectively) at the freezing temperature. No harmful effects have ever been noted due to the presence of these elements in these concentrations and therefore analyses are not required for these elements.

*New Section.*—Add the following new Section 2 entitled "Basis of Purchase" and renumber the subsequent sections accordingly:

2. Orders for ingot under these specifications shall include the following information:
(*1*) Quantity in pounds,
(*2*) Alloy (see Table I), and
(*3*) Size, if not vendor's standard.

## REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

**Standard Specification for Magnesium-Base Alloy Die Castings (B 94 - 52):**[2]

*New Section.*—Add a new Section 2 "Basis of Purchase" as recommended above for Specifications B 85 and renumber the subsequent sections accordingly.

*Table I.*—Delete alloy AM100B. For alloys AZ91A and AZ91B, change the zinc limits from "0.4 to 1.0" to read "0.35 to 1.0," and change the maximum silicon content from "0.5" to read "0.50." For alloy AZ91B, change the maximum

copper content from "0.3" to read "0.35."

*New Section.*—Add the following new Section 8 entitled "Mechanical Properties and Tests."

8. Unless specified in the order or specifically guaranteed by the manufacturer, acceptance of die castings under these specifications shall not depend on mechanical properties determined by tension or impact tests. When tension or impact tests are made, the test specimens and procedures shall be as prescribed in Explanatory Note 1.

*Explanatory Notes.*—Delete all reference to alloy AM100B. In Note 1, the second sentence, change "physical properties" to read "mechanical properties," and delete the third sentence.

**Standard Specification for Copper-Base (Brass) Alloy Die Castings (B 176 – 52):**[2]

*New Section.*—Add a new Section 2 "Basis of Purchase" as recommended above for Specifications B 85, and renumber the subsequent sections accordingly.

*Section 4.*—In the heading and in line 5, change "physical properties" to read "mechanical properties."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Aluminum-Base Die-Casting Alloys* (E. V. Blackmun, chairman) is testing the last of the aluminum alloys (G8A) in connection with the revision of the typical mechanical properties. Data for all of the alloys should be ready for study in June.

The subcommittee prepared the revisions in Specification B 85 – 56 T covered earlier in this report.

*Subcommittee II on Zinc-Base Die-Castings Alloys* (A. E. Weiss, chairman) prepared the revisions in Specifications B 86 – 53 T and B 240 – 52 T covered earlier in this report.

A task group is drafting a specification covering three alloys used as hardeners in the preparation of the two zinc die casting alloys.

*Subcommittee V on Exposure and Corrosion Tests* (D. H. Kleppinger, chairman) inspected the exposure site on the roof of the New York Port Authority Building and found no evidence of contamination of specimens with oil, grease, or other dirt which could be attributed to use of the adjacent space for helicopter operation. Therefore, the exposure of the SC84A controlled zinc bars will be continued at this site.

A progress report on the Exposure Tests on SC84A Aluminum Bars with Varying Zinc Contents, prepared by Messrs. Colwell, Blackmun, and Kleppinger, is appended hereto.[4]

Zinc and magnesium alloy test bars will be removed from exposure sites in 1959 after exposure for 20 years.

*Subcommittee VII on Magnesium-Base Die-Casting Alloys* (V. D. Sweeney, chairman) prepared the revisions in Specification B 94 – 52 covered earlier in this report.

A task group collecting typical properties for magnesium die castings has submitted data for study.

*Subcommittee VIII on Brass Die-Casting Alloys* (J. C. Fox, chairman) prepared the revisions in Specification B 176 – 52 covered earlier in this report.

*Subcommittee IX on Die-Casting Processes* (A. E. Martin, chairman) has completed its assignment and is discharged with thanks.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] See p. 174.

This report has been submitted to letter ballot of the committee, which consists of 92 members: 86 members returned their ballots, of whom 79 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. BABINGTON,
*Chairman.*

GEORGE L. WERLEY,
*Secretary.*

## APPENDIX

# EXPOSURE TESTS OF SC84A ALUMINUM BARS WITH VARYING ZINC CONTENT

## PROGRESS REPORT OF TASK GROUP OF SUBCOMMITTEE V ON EXPOSURE AND CORROSION TESTS

At the June, 1949, meeting of Committee B-6 a proposal was made to increase the zinc content of the SC84A alloy from 0.6 to 1.0 per cent, but before approving this change the committee decided to make tests on SC84A with varying zinc contents. The five zinc contents chosen for study were 0.25, 0.50, 1.0, 1.5, and 2.0 per cent, and the results of the mechanical testing were given in Appendix I to the 1953 report of Committee B-6.[1]

At the 1953 meeting of Committee B-6, a new series of exposure tests on the same set of test bars was initiated as the total quantity of bars had not been used in the mechanical tests. Bars were exposed, therefore, at the 80- and 800-ft test sites at Kure Beach, N. C., and New York City, roof of Port Authority Building, for 1-, 3-, 6-, and 12-yr periods. A test site at Columbus, Ohio, was also originally chosen, but through a misunderstanding these bars were never exposed.[1] The bars were taken from storage in April, 1953, and in April, 1954, the 1-yr test bars from New York City and Kure Beach were withdrawn and tested and reported to the Society at the 1955 meeting.[2]

In April, 1956, the 3-yr bars were removed and tested and the results were reported to Subcommittee V at the June, 1956, meeting, and this task group was authorized to prepare a report to the Society on the results obtained. In the removal of the test bars it was found that all but 19 of the 80 bars at the 80-ft site at Kure Beach had been lost in Hurricane Hazel on October 15, 1954. The lost bars were not replaced, and therefore the 3-yr results at the 80-ft site are incomplete.

The test bars were made by three producers: Precision Castings Co., Stewart Warner Co., and the Bell Telephone Laboratories. All bars were made from a single master heat of metal produced by the Apex Smelting Co. in five lots, each lot having a different zinc content. One producer also made bars with approximately 3 per cent zinc. The bars were tested by the Bell Telephone Laboratories, the American Smelting and Refining Co. Laboratories, and the Pitman-Dunn Laboratories of Frankford Arsenal. The individual results were consistent enough to be averaged, so that in this report each figure represents the average of five bars each from three producers, each tested by three laboratories, or a total of 45 bars (except in a few instances where bars were lost).

The composition of the ingot used by

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[1] Report of Committee B-6 on Die Cast Metals and Alloys, Appendix I, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 212 (1953).

[2] Report of Committee B-6 on Die Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 246 (1955).

all is given in Table I, and analyses of the resulting test bars showed no significant change in composition during the casting operation.[1]

The average mechanical properties in the as-cast condition are shown in Table II,[1, 2] and the mechanical properties after the first year exposure are shown in Table III.[2]

TABLE I.—COMPOSITION OF INGOT USED BY ALL.

| Alloy (per cent Zinc) | Composition, per cent | | | | | | |
|---|---|---|---|---|---|---|---|
| | Copper | Iron | Magnesium | Manganese | Nickel | Silicon | Zinc |
| 1 (0.25) | 3.52 | 0.56 | 0.02 | 0.45 | 0.15 | 8.64 | 0.19 |
| 2 (0.50) | 3.42 | 0.58 | 0.02 | 0.46 | 0.17 | 8.61 | 0.47 |
| 3 (1.0) | 3.43 | 0.58 | 0.02 | 0.45 | 0.16 | 8.76 | 1.01 |
| 4 (1.5) | 3.38 | 0.57 | 0.02 | 0.43 | 0.16 | 8.42 | 1.55 |
| 5 (2.0) | 3.45 | 0.59 | 0.02 | 0.46 | 0.16 | 8.41 | 1.99 |
| 6 (3.0) | (Same as No. 5 with Zn increased to 3.0 per cent) | | | | | | |

TABLE II.—MECHANICAL PROPERTIES—AS CAST.

(Average* of Producers A, B, and C and Laboratories X, Y, and Z.)

| Alloy (per cent Zinc) | Tensile Strength, psi | Yield Strength, psi | Per cent Elongation |
|---|---|---|---|
| 1 (0.25) | 41 700 | 20 400 | 3.4 |
| 2 (0.50) | 41 500 | 20 100 | 3.5 |
| 3 (1.0) | 41 900 | 20 100 | 3.7 |
| 4 (1.5) | 42 100 | 20 200 | 3.8 |
| 5 (2.0) | 42 600 | 20 200 | 4.0 |
| 6*(3.0) | 41 800 | 20 200 | 3.9 |

* Except No. 6, from Producer B only.

The mechanical properties from Table III have been plotted in Fig. 1 which shows graphically that none of the three properties changed with variation in zinc content. The corrosion at the two Kure Beach sites for 1 yr caused lower tensile strengths and elongations, and there was no significant difference between the 80-ft and the 800-ft sites. The tensile strength was not affected at New York, but the elongation dropped somewhat due to aging of the material. The yield strengths at all three sites increased by the same amounts, an effect due to aging rather than to corrosion.

The mechanical properties after 3-yr exposure are shown in Table IV. Many of the 80-ft Kure Beach bars are missing entirely and the others represent only a partial number of bars. These values have been plotted in Fig. 2 and the conclusions after 3-yr exposure are the same as those for 1 yr.

It was thought desirable also to present the changes in mechanical properties due to exposure by showing the percentage of change, and these values for 1 yr are presented in Table V, and for 3-yr exposure in Table VI. The percentage of changes are again shown graphically in Fig. 3, the light bars illustrating 3-yr exposure and the dark bars 1-yr exposure. Increases in yield strength and decreases in elongation are reasonably constant and are largely due to aging. Changes in tensile strength at New York are insignificant; at the two Kure Beach sites results show a drop in tensile strength due to corrosion. There might be some indication of increasing corrosion loss with increasing zinc, at the two Kure Beach sites, but the trend is so slight that such a conclusion cannot be made positively. For example, at the 800-ft site the 0.25 zinc bars lost 41 per cent of their elongation after 1 yr, and the 2 per cent zinc bars lost 52 per cent of their elongation after 3 yr. The conclusion is believed justified, therefore, that within the time limita-

TABLE III.—MECHANICAL PROPERTIES AFTER ONE-YEAR EXPOSURE.
(Average* of Producers A, B, and C and Laboratories X, Y, and Z.)

| Alloy | Tensile Strength, psi | | | Yield Strength, psi | | | Per Cent Elongation | | |
|---|---|---|---|---|---|---|---|---|---|
| | New York | Kure Beach 80 | Kure Beach 800 | New York | Kure Beach 80 | Kure Beach 800 | New York | Kure Beach 80 | Kure Beach 800 |
| 1 | 42 500 | 37 400 | 38 500 | 23 600 | 23 500 | 23 200 | 2.9 | 1.9 | 2.0 |
| 2 | 40 800 | 36 200 | 38 000 | 23 400 | 22 900 | 23 100 | 2.8 | 1.9 | 1.9 |
| 3 | 42 400 | 36 100 | 36 600 | 23 600 | 22 900 | 23 400 | 3.1 | 1.8 | 2.1 |
| 4 | 42 000 | 36 200 | 37 400 | 23 400 | 23 100 | 22 900 | 3.0 | 1.8 | 2.0 |
| 5 | 41 700 | 36 900 | 36 800 | 23 200 | 23 300 | 23 000 | 3.0 | 2.0 | 1.8 |
| 6* | 42 500 | 35 300 | 36 500 | 23 900 | 23 300 | 23 200 | 3.1 | 1.9 | 1.9 |

* Except No. 6 from Producer B only, and except for some missing bars.

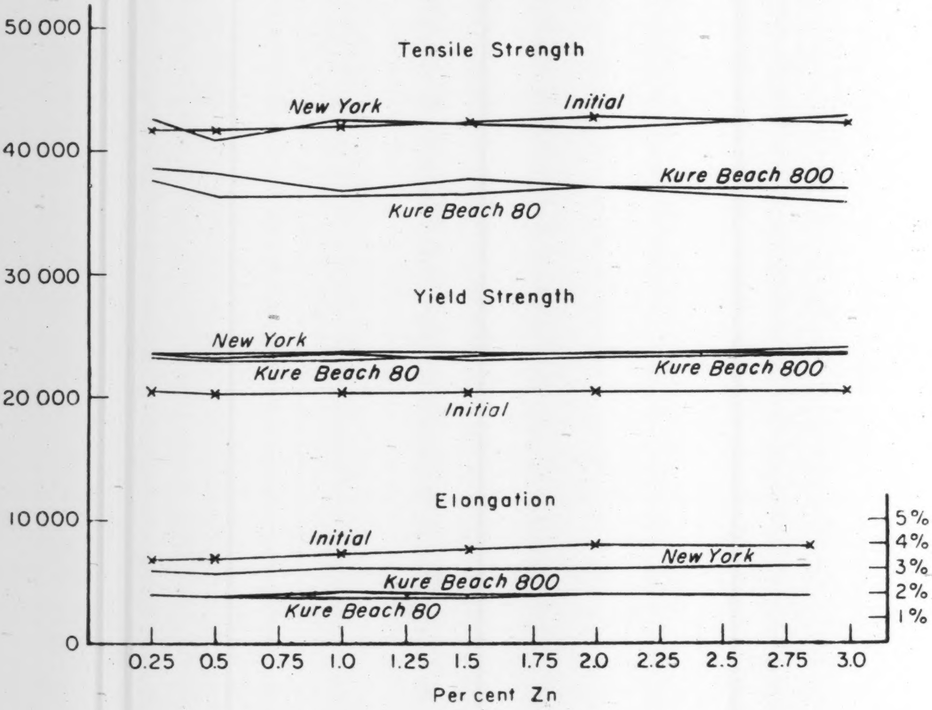

FIG. 1.—SC84A with Increasing Zinc after 1-yr Exposure at Three Sites.

TABLE IV.—MECHANICAL PROPERTIES AFTER THREE-YEAR EXPOSURE.
(Average* of Producers A, B, and C and Laboratories X, Y, and Z.)

| Alloy | Tensile Strength, psi | | | Yield Strength, psi | | | Per Cent Elongation | | |
|---|---|---|---|---|---|---|---|---|---|
| | New York | Kure Beach 80 | Kure Beach 800 | New York | Kure Beach 80 | Kure Beach 800 | New York | Kure Beach 80 | Kure Beach 800 |
| 1 | 41 600 | 36 900 | 37 700 | 27 500 | 24 800 | 23 600 | 2.3 | 1.8 | 1.9 |
| 2 | 39 900 | 39 000 | 37 300 | 23 500 | 24 700 | 23 000 | 2.3 | 2.1 | 2.0 |
| 3 | 42 400 | * | 37 100 | 25 100 | * | 23 100 | 3.1 | * | 2.0 |
| 4 | 40 200 | 35 400 | 36 200 | 23 400 | 22 900 | 23 100 | 2.5 | 2.0 | 1.8 |
| 5 | 41 500 | * | 36 600 | 24 500 | * | 23 200 | 2.6 | * | 1.9 |
| 6* | * | * | 35 900 | * | * | 23 200 | * | * | 1.7 |

* Except No. 6 from Producer B only, and except for missing bars from Hurricane Hazel.

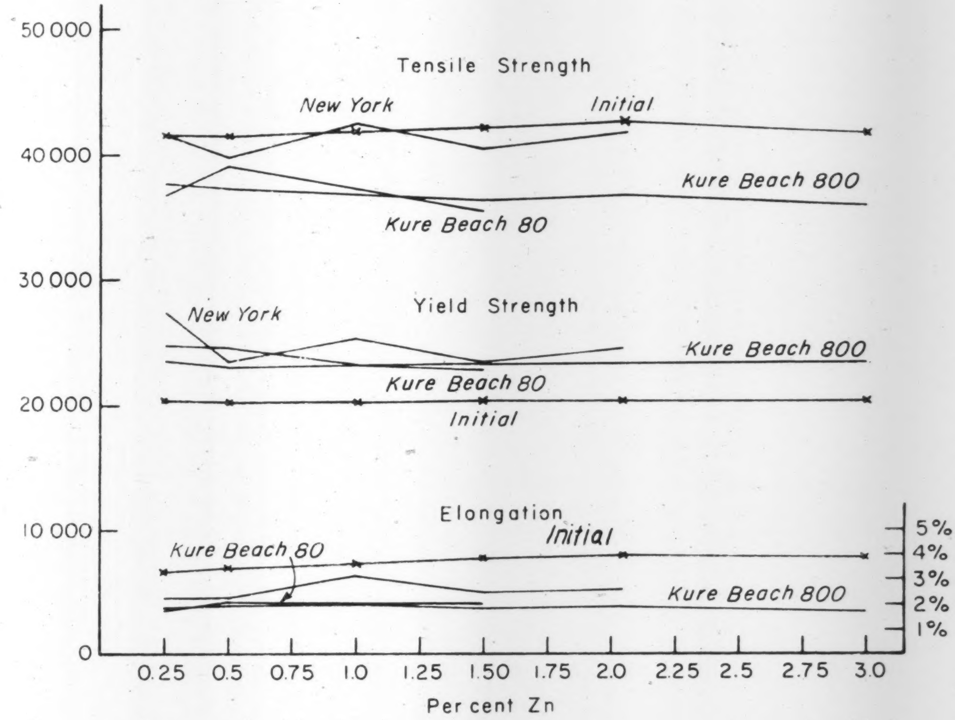

FIG. 2.—SC84A with Increasing Zinc after 3-yr Exposure at Three Sites.

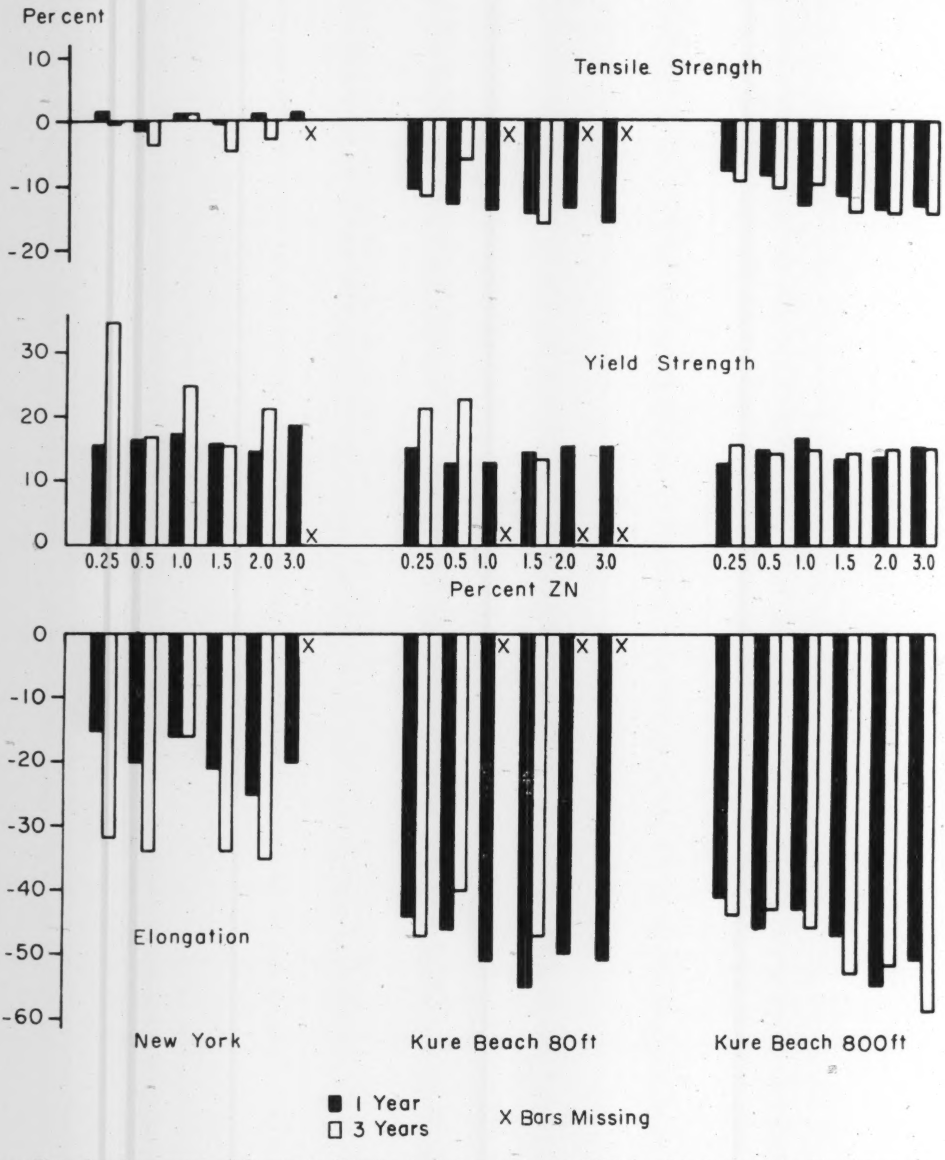

FIG. 3.—Percentage Change in Mechanical Properties of SC84A with Increasing Zinc after 1-yr and 3-yr Exposure.

tions of this test, the corrosion resistance of SC84A is not affected by zinc contents up to 2 per cent or more.

The 3-yr exposure completes the test at the Kure Beach 80-ft site. One set remains at the 800-ft site at Kure Beach for a 6-yr exposure, and two sets remain at the New York Port Authority

TABLE V.—PER CENT CHANGES IN MECHANICAL PROPERTIES.
After one year [(Table II − Table III) ÷ Table II] × 100 per cent.

| Alloy | Tensile Strength | | | Yield Strength | | | Elongation | | |
|---|---|---|---|---|---|---|---|---|---|
| | New York | Kure Beach 80 | Kure Beach 800 | New York | Kure Beach 80 | Kure Beach 800 | New York | Kure Beach 80 | Kure Beach 800 |
| 1 | +1.9 | −10.3 | −7.7 | +15.7 | +15.2 | +12.7 | −15 | −44 | −41 |
| 2 | −1.7 | −12.8 | −8.2 | +16.4 | +12.9 | +14.9 | −20 | −46 | −46 |
| 3 | +1.2 | −13.8 | −12.7 | +17.4 | +12.9 | +16.4 | −16 | −51 | −43 |
| 4 | −0.2 | −14.1 | −11.2 | +15.9 | +14.4 | +13.4 | −21 | −53 | −47 |
| 5 | +1.2 | −13.4 | −13.6 | +14.9 | +15.4 | +13.9 | −25 | −50 | −55 |
| 6 | +1.7 | −15.5 | −12.7 | +18.3 | +15.4 | +14.9 | −20 | −51 | −51 |

TABLE VI.—PER CENT CHANGES IN MECHANICAL PROPERTIES.
After three years [(Table II − Table IV) ÷ Table II] × 100 per cent.

| Alloy | Tensile Strength | | | Yield Strength | | | Elongation | | |
|---|---|---|---|---|---|---|---|---|---|
| | New York | Kure Beach 80 | Kure Beach 800 | New York | Kure Beach 80 | Kure Beach 800 | New York | Kure Beach 80 | Kure Beach 800 |
| 1 | −0.2 | −11.5 | −9.6 | +34.8 | +21.6 | +15.7 | −32 | −47 | −44 |
| 2 | −3.9 | −6.0 | −10.1 | +16.9 | +22.8 | +14.4 | −34 | −40 | −43 |
| 3 | +1.2 | * | −10.0 | +24.9 | * | +14.9 | −16 | * | −46 |
| 4 | −4.5 | −15.9 | −14.0 | +15.8 | +13.4 | +14.3 | −34 | −47 | −53 |
| 5 | −2.6 | * | −14.1 | +21.3 | * | +14.8 | −35 | * | −52 |
| 6 | * | * | −14.1 | * | * | +14.8 | * | * | −59 |

* Bars lost in Hurricane Hazel. A few of the others are not complete due to partial loss.

roof for 6- and 12-yr exposures. In addition to this, the bars originally scheduled for Columbus have been retained at Frankford Arsenal for testing as controls at the termination of the program after the 12-yr exposure. The correction of the percentage of changes shown in Tables V and VI by the changes due solely to aging will clarify some of these results on the effects of corrosion. When this test was originally planned it was felt that corrosion, particularly at Kure Beach, would be much more severe, and that after 3 yr at the 80-ft site the bars would be largely destroyed. This has proved not to be the case, and therefore the normal aging effect has a greater significance than was considered in the original plans.

Respectfully submitted by the Task Group,

D. L. Colwell, *Chairman.*
E. V. Blackmun and
D. H. Kleppinger.

# REPORT OF COMMITTEE B-7

## ON

# LIGHT METALS AND ALLOYS, CAST AND WROUGHT*

Committee B-7 on Light Metals and Alloys held two meetings during the year: at Atlantic City, N. J., in June, 1956, and at Philadelphia, Pa., in February, 1957.

The committee consists of 85 members, of whom 82 are voting members, 37 are classified as producers, 30 as consumers, and 18 as general interest members.

P. V. Faragher resigned as chairman of Subcommittee III because of retirement and was replaced by H. D. Monsch; R. B. Smith resigned as chairman of Subcommittee VII and was replaced by C. B. Gleason; J. C. Jones passed away and H. M. Garner resigned from the committee because of retirement.

A new Subcommittee IX (Editorial) is being organized.

The American Standards Assn. which represents the U. S. in the International Organization for Standardization (ISO) has become a participating member of Technical Committee 79 on Light Metals and Their Alloys, and has designated Committee B-7 as its advisory group.

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, the committee presented to the Society through the Administrative Committee on Standards a new Tentative Specification for Round Aluminum-Alloy Welded Tubes. The specification was accepted by the administrative committee on September 5, 1956, and it appears in the 1956 Supplement to Book of ASTM Standards, Part 2, bearing the designation B 313 - 56 T.

### RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends three new specifications for publication as tentative, revisions in 21 tentative specifications, revisions for immediate adoption in 3 standard specifications, withdrawal of 3 tentative specifications, and an editorial change in one standard. The standards and tentatives affected, together with the revisions recommended, are covered in detail in the Appendix to this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Aluminum and Aluminum Alloy Ingots* (R. A. Quadt, chairman) prepared revisions of the Standard Specifications for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 56), including the addition of requirements referring to packing, marking, and shipping; and revision of chemical requirements in the Standard Specifications for Aluminum for Use in Iron and Steel Manufacture (B 37 - 49), including additional requirements referring to packing, marking, and shipping. All changes are set forth in the Appendix to this report.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16-21, 1957.

[1] See p. 183.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee II on Aluminum Alloy Castings* (D. L. Colwell, chairman) prepared the revision of Tentative Specification for Aluminum-Base Alloy for Sand Castings (B 26 – 56 T) and Tentative Specification for Aluminum-Base Permanent Mold Castings (B 108 – 56 T) as set forth in the Appendix to this report. These changes add a section on packing, marking, and shipping to both specifications and in B 26 increase the maximum magnesium allowance in alloy CS43A from 0.05 per cent to 0.10 per cent max.

*Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys* (H. D. Monsch, chairman) prepared the new Tentative Specification for Aluminum-Alloy Rivet and Cold Heading Wire and Rods, the new Tentative Specification for Type A and for Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes, the new Tentative Specification for Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors),[3] and revisions as set forth in the Appendix to this report covering a new section on packing, marking, and shipping of Tentative Specifications B 209, B 210, B 211, B 221, B 234, B 235, B 236, B 241, B 247, B 307, B 308, and B 313.

In addition, changes are recommended in B 209 to incorporate the requirements of B 178 – 56 T by adding chemical requirements and tensile requirements for alloys 996A, G1B, GM41A, and GM51A and dimensional tolerances to cover thickness variation in wide widths of light-gage sheet, variation in width of coiled sheet, and variation in width of light-gage flat sheet.

Changes in Specifications B 211, B 221, and B 308 are recommended to incorporate requirements of Specification B 273; to add chemical requirements and tensile requirements for alloy 996A to B 211, also dimensional tolerances for length, straightness, and squareness of cut ends of rolled or cold-finished rods and bar; to add chemical requirements and tensile requirements for alloy GM51A to B 221, also dimensional tolerances for variation in angularity, length, flatness, contour, depth of surface defects, and squareness of cut ends of extruded bars, rods, and shapes; also to add chemical requirements and tensile requirements for alloys GM51A and GS11C, and tensile requirements for the T62 temper of alloy GS11A to B 308.

Changes in Specifications B 210, B 235, and B 241 are recommended to incorporate requirements of Specification B 274; to add chemical requirements and tensile requirements for alloys 996A, GR40A, GS10A, and Clad M1A to Specification B 210, also dimensional tolerances for corner radii, straightness, degree of twist, length, flatness for other than round tube, squareness of cut ends, and nominal angle for other than round tube; to Specification B 235, chemical requirements and tensile requirements for alloys 996A, GR20A, Clad M1A, and MG11A in dimensional tolerances for length, corner radii for tube other than round, straightness, degree of twist for tube other than round, squareness of cut ends, and nominal angle for other than round tube; to Specification B 241 to add the permissible variations in dimensions and weight for ASA schedules 5 and 10.

The revisions in Specification B 234, in addition to adding a new section, consist of adding chemical requirements for alloy 996A, tensile requirements for alloys 996A and Clad M1A, and revision of the flattening test to provide for addition of alloys GS11A and GS11C and revision of the section Cladding Thickness to add requirements for cladding on the outside surface only, and editorial corrections in Tables I, II, III, and VI.

The revisions in Specification B 247,

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[3] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 2.

in addition to adding a new section, increased the thickness limit to which the tensile requirements are applicable for alloys identified by note sign *e* in Table II.

*Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought* (A. A. Moore, chairman) revised Specification B 92 – 56 and Tentative Specifications B 80, B 90, B 91, B 93, B 107, B 199, and B 217 as set forth in the Appendix of this report. These revisions include extensive editorial changes and addition of packaging and marking sections to all the specifications. The minimum tensile yield strength of ZE41A-T5 was increased and the elongation requirement of the same material decreased in B 80, and a new Table of properties and characteristics added.

Alloys AZ31B, AZ31C and HK31A sheet and plate were added to B 90, and the dimensional tolerances were changed and expanded to cover plate. Alloy AZ31C was added to B 107, and minimum properties of some of the alloys and conditions revised upward. Minimum properties were added also for larger size categories. Alloys AZ91C, EK41A and EZ33A were added to B 199. A Table of properties and characteristics was added for all alloys now in B 199. Specification B 217 was revised to include Alloy AZ31C.

*Subcommittee V on Testing Light Metals* (J. C. Millson, chairman) is investigating statistical sampling methods as applied to testing for mechanical properties, and in cooperation with Committee E-11 on Quality Control of Materials, is attempting to establish lot sizes to be used in defining a statistically satisfactory sampling frequency and method for application to the tensile requirements in specifications under the jurisdiction of Committee B-7.

A task group is considering suitable testing speeds for short-time high-temperature tension tests of light metals.

A task group is determining the satisfactory minimum size of subsize tensile samples.

*Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium Alloys* (Fred Keller, chairman) is preparing proposed tentative specifications for anodic coatings on aluminum and aluminum alloys.

*Subcommittee VII on Codification of Light Metals and Alloys, Cast and Wrought* (C. B. Gleason, chairman) confined its activities to assigning proper designations for new alloys, and in maintaining records of those previously assigned in order to prevent duplication or misapplication.

*Subcommittee VIII on Atmospheric Exposure Tests* (L. H. Adam, chairman) has specimens of wrought and cast, aluminum and magnesium alloys installed at five ASTM exposure test sites. Current data on material exposed for three years are being evaluated for report to the committee.

This report has been submitted to a letter ballot of the committee, which consists of 85 members; 54½ members returned their ballots, of whom 54½ have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

I. V. WILLIAMS,
*Chairman.*

R. B. SMITH,
*Secretary.*

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-7 presented to the Society through the Administration Committee on Standards revisions of Tentative Specification for Aluminum-Alloy Round Welded Tubes (B 313 – 57 T). The revisions were accepted by the Standards Committee on November 1, 1957, and the revised specification appears in the 1957 Supplement to Book of ASTM Standards, Part 2.

## APPENDIX

# PROPOSED RECOMMENDATIONS AFFECTING STANDARDS FOR LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering light metals and alloys which are referred to earlier in this report. The standards appear in their present form in the 1955 Book of ASTM Standards, Part 2, or the 1956 Supplement to Book of ASTM Standards, Part 2.

In all the specifications under the jurisdiction of Committee B-7, it is recommended that the word "specifications" be changed to the singular "specification" in the titles, scopes, and throughout the text.

## NEW TENTATIVES

The committee recommends that the following three specifications be accepted for publication as tentative as appended hereto:[1]

*Tentative Specifications for:*

Aluminum-Alloy Rivet and Cold Heading Wire and Rods,

Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes, for Electrical Purposes (Bus Conductors), and

Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes.

## REVISION OF TENTATIVES

The committee recommends that the following 21 tentative specifications be revised as indicated below and continued as tentative:

### Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 56 T):

*New Section.*—Add a new Section 13 titled "Packing, Marking and Shipping" to read as follows, renumbering all subsequent sections accordingly:

13. (*a*) The material shall be packed in such a manner as to prevent damage in ordinary handling and transportation. The type of packing and gross weight of individual containers shall be left to the discretion of the manufacturer unless otherwise agreed upon. Packing methods and containers shall be so selected as to permit maximum utility of mechanical equipment in unloading and subsequent handling. Each package or container shall contain only one size, alloy or condition of material when packed for shipment unless otherwise agreed upon.

(*b*) Each package or container shall be marked with the purchase order number, drawing number, quantity, specification number, alloy and condition, gross and net weights, and the name of the manufacturer.

(*c*) Packages or containers shall be such as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

*Table I.*—For alloy CS43A, revise the magnesium limit from the present "0.05" to read "0.10" per cent.

### Tentative Specification for Magnesium-Base Alloy Sand Castings (B 80 – 56 T):

*Section 3.*—Revise the title to read "Materials and Manufacture."

*New Section.*—Add a new Section 12 titled "Retests" to read as follows, renumbering subsequent sections accordingly:

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[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 2.

12. If the results of the tension tests do not conform to the requirements prescribed in Table II, the castings may be reheat-treated but not more than twice. The results of acceptable tests shall conform to the requirements as to tensile properties specified in Table II.

TABLE I.—PROPERTIES AND CHARACTERISTICS.
(Revision of Table IV, B 80 – 56 T)

| Alloy | Melting Range, Approximate deg Fahr | | | Pattern Shrinkage Allowance, in. per ft[b] | Foundry Characteristics[c] | | | | Other Characteristics[c] | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Non-Equilibrium Solidus[a] | Solidus | Liquidus | | Pressure Tightness | Fluidity[d] | Microporosity Tendency[e] | Normally Heat Treated | Machining[f] | Electroplating[g] | Surface Treatment[h] | Suitability to Brazing[i] | Suitability to Welding[j] |
| AM100A...... | 810 | 867 | 1100 | 5/32 | 2 | 1 | 2 | Yes | 1 | 2 | 2 | No | 1 |
| AZ63A....... | 685 | 850 | 1130 | 5/32 | 3 | 1 | 3 | Yes | 1 | 1 | 1 | No | 3 |
| AZ81A....... | 790 | 882 | 1115 | 5/32 | 2 | 1 | 2 | Yes | 1 | 2 | 2 | No | 1 |
| AZ91C....... | 785 | 875 | 1105 | 5/32 | 2 | 1 | 2 | Yes | 1 | 2 | 2 | No | 2 |
| AZ92A....... | 770 | 830 | 1100 | 5/32 | 2 | 1 | 2 | Yes | 1 | 2 | 2 | No | 2 |
| EK30A....... | ... | 1100 | 1184 | 7/32 | 1 | 2 | 1 | Yes | 1 | 2 | 1 | No | 1 |
| EK41A....... | ... | 1095 | 1193 | 7/32 | 1 | 2 | 1 | Yes | 1 | 2 | 1 | No | 1 |
| EZ33A....... | ... | 1010 | 1189 | 3/16 | 1 | 2 | 1 | Yes | 1 | 2 | 1 | No | 1 |
| HK31A....... | ... | 1092 | 1204 | 7/32 | 1 | 2 | 1 | Yes | 1 | 2 | 1 | [k] | 1 |
| HZ32A....... | ... | 1026 | 1198 | 3/16 | 1 | 2 | 1 | Yes | 1 | [k] | 2 | [k] | 2 |
| ZE41A....... | ... | ... | ... | ... | [k] | 2 | [k] | Yes | 1 | 1 | 1 | No | [k] |
| ZH62A....... | ... | ... | 1169 | 5/32 | 2 | 2 | 2 | Yes | 1 | 1 | 1 | No | [k] |
| ZK51A....... | ... | 1020 | 1185 | 5/32 | 3 | 2 | 2 | Yes | 1 | 2 | 1 | No | 3 |
| ZK61A....... | ... | 985 | 1175 | 5/32 | 3 | 2 | 2 | Yes | 1 | 2 | 1 | No | 3 |

[a] As measured on metal solidified under normal casting conditions.

[b] Allowance for average castings. Shrinkage requirements will vary with intricacy of design and dimensions.

[c] Rating of 1 indicates best of group; 3 indicates poorest of group.

[d] Ability of liquid alloy to flow readily in mold and fill thin sections.

[e] Based on radiographic evidence.

[f] Composite rating based on ease of cutting, chip characteristics, quality of finish, and tool life. Ratings, in the case of heat-treatable alloys, based on T6 type temper. Other tempers, particularly the annealed temper, may have lower rating.

[g] Ability of casting to take and hold an electroplate applied by present standard methods.

[h] Ability of castings to be cleaned in standard pickle solutions and to be conditioned for best paint adhesion.

[i] Refers to suitability of alloy to withstand brazing temperature without excessive distortion or melting.

[j] Based on ability of material to be fusion-welded with filler rod of same alloy.

[k] Inexperience with these alloys under wide production conditions makes it undesirable to supply ratings at this time.

*New Section.*—Add a new Section 14 as indicated above for new Section 13 in Specification B 26, renumbering subsequent sections accordingly.

*Table I.*—For alloys HZ32A and ZH62A change the zirconium requirement from the present "0.50" per cent minimum to read "0.50 to 1.0" per cent. For alloy EZ33A change the zinc requirement from the present "2.0 to 4.0" to read "2.0 to 3.1" per cent.

*Table II.*—For alloy ZE41A-T5 raise the minimum yield strength from the present "16,000" to read "19,000" psi, and decrease the minimum elongation requirement from the present "4" to read "2.5" per cent.

*Table III.*—For alloys AM100A-T6, AZ63A-T6, AZ91C-T6, AZ92A-T6, and ZK61A-T6 delete the typical yield strength and corresponding extension

under load values. For alloy ZE41A-T5 increase the minimum yield strength from the present "16,000" to read "19,000" psi and increase the extension under load value from the present "0.0045" to read "0.0049" in. per in.

*Table IV.*—Revise to read as shown in the accompanying Table I.

*Explanatory Note 1.*—For alloy HZ32A revise the last sentence to read "It is recommended for elevated temperature use up to 650 F. However, each application should be thoroughly reviewed for conditions of time, temperature, and stress, because magnesium alloys exhibit a loss of mechanical properties when exposed to elevated temperatures."

### Tentative Specification for Magnesium-Base Alloy Sheet (B 90 – 56 T):

This specification has been completely revised and is appended hereto in its revised form.[2]

### Tentative Specification for Magnesium-Base Alloy Forgings (B 91 – 56 T):

*Section 3.*—Revise the title to read "Materials and Manufacture."

*New Section.*—Add a new Section 11 titled "Retests" to read as follows, renumbering subsequent sections accordingly:

> 11. If the results of the tension tests do not conform to the requirements prescribed in Table II, the forgings may be reheat-treated but not more than twice. The results of acceptable tests shall conform to the requirements of the tensile properties specified in Table II.

*New Section.*—Add a new Section 13 as indicated above for new Section 13 in Specification B 26, renumbering subsequent sections accordingly.

*Table II.*—In the first column revise "Alloy" to read "Alloy and Condition."

### Tentative Specification for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 – 56 T):

*Section 2.*—Add an Item (*4*) to read "Place of Inspection (Section 9 (*a*))."

*Section 3.*—Change the title to read "Materials and Manufacture" and revise to read as follows:

> 3. The material may be made by any approved process. The materials used shall be such as to produce products that will conform to the requirements as to chemical composition prescribed in this specification.

*New Section.*—Add a new Section 7 titled "Workmanship and Finish" to read "The ingots shall be uniform in quality and shall be commercially free from slag or other foreign material" and renumber subsequent sections accordingly.

*New Section.*—Add a new Section 8 to read as indicated above for new Section 13 in Specification B 26, renumbering subsequent sections accordingly.

*New Section.*—Add a new Section 9 titled "Inspection" to read as follows, renumbering subsequent sections accordingly:

> 9. (*a*) Inspection may be made at the manufacturer's works where the castings are made, or at the point at which they are received, at the option of the purchaser.
>
> (*b*) If the purchaser elects to have the inspection made at the manufacturer's works, the manufacturer shall afford the inspector representing the purchaser all reasonable facilities, without charge, to satisfy him that the material is being furnished in accordance with this specification. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

*Table I.*—Add Notes 1 and 2 to read as follows:

> NOTE 1.—Analysis shall regularly be made only for the elements specifically mentioned in this table. If, however, the presence of other elements is suspected or indicated in the course of routine analysis, further analysis shall be made to determine that the total of these other elements is not in excess of the limits specified in the last column of the table.

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[2] The revised tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 2.

## TABLE II.—TENSILE REQUIREMENTS.

(Revision of Table II, B 107 – 56 T)

NOTE.—For purposes of determining conformance with this specification, each value for tensile strength and yield strength shall be rounded off to the nearest 100 psi, and each value for elongation shall be rounded off to the nearest 0.5 per cent, both in accordance with the rounding-off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E 29).[3]

| Alloy and Condition | Form | Diameter or Thickness, in.[a] | Cross-Sectional Area, sq in. | Tensile Strength, min, psi | Yield Strength (0.2 per cent offset), min, psi | Elongation in 2 in., min, per cent |
|---|---|---|---|---|---|---|
| AZ31B-F | Bars, rods, and shapes | 0.249 and under | All | 35 000 | 21 000 | 7 |
| | | 0.250 to 1.499 | All | 35 000 | 22 000 | 7 |
| | | 1.500 to 2.499 | All | 34 000 | 22 000 | 7 |
| | | 2.500 to 4.999 | All | 32 000 | 20 000 | 7 |
| | Hollow shapes | All | All | 32 000 | 16 000 | 8 |
| AZ61A-F | Bars, rods, and shapes | 0.249 and under | All | 38 000 | 21 000 | 8 |
| | | 0.250 to 2.499 | All | 39 000 | 24 000 | 9 |
| | | 2.500 to 4.999 | All | 40 000 | 22 000 | 7 |
| | Hollow shapes | All | All | 36 000 | 16 000 | 7 |
| AZ80A-F | Bars, rods and shapes | 0.249 and under | All | 43 000 | 28 000 | 9 |
| | | 0.250 to 1.499 | All | 43 000 | 28 000 | 8 |
| | | 1.500 to 2.499 | All | 43 000 | 28 000 | 6 |
| | | 2.500 to 4.999 | All | 42 000 | 27 000 | 4 |
| AZ80A-T51 | Bars, rods, and shapes | 0.249 and under | All | 47 000 | 30 000 | 4 |
| | | 0.250 to 2.499 | All | 48 000 | 33 000 | 4 |
| | | 2.500 to 4.999 | All | 45 000 | 30 000 | 2 |
| M1A-F | Bars, rods, and shapes | 0.249 and under | All | 30 000 | [b] | 2 |
| | | 0.250 to 1.499 | All | 32 000 | [b] | 3 |
| | | 1.500 to 2.499 | All | 32 000 | [b] | 2 |
| | | 2.500 to 4.999 | All | 29 000 | [b] | 2 |
| | Hollow shapes | All | All | 28 000 | [b] | 2 |
| ZK60A-F | Bars, rods, and shapes | All | 4.999 and under | 43 000 | 31 000 | 5 |
| | | All | 5.000 to 39.999 | 43 000 | 31 000 | 4 |
| | Hollow shapes | All | All | 40 000 | 28 000 | 5 |
| ZK60A-T5 | Bars, rods, and shapes | All | 4.999 and under | 45 000 | 36 000 | 4 |
| | Hollow shapes | All | All | 46 000 | 38 000 | 4 |

[a] Intermediate dimensions shall be rounded off to the third decimal place in accordance with the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E 29).[3]

[b] Not required.

NOTE 2.—The following applies to all specified limits in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit.

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[3] 1955 Book of ASTM Standards, Part 2.

TABLE III. MINIMUM COMPRESSIVE YIELD STRENGTH VALUES.
(Revision of Table X, B 107 – 56 T)

| Alloy and Condition | Form | Cross-Sectional Area, sq in. | Compressive Yield Strength, min, psi |
|---|---|---|---|
| ZK60A-F | Bars, rods, and shapes | 1.999 and under | 27 000 |
| | | 2.000 to 2.999 | 26 000 |
| | | 3.000 to 4.999 | 25 000 |
| | | 5.000 to 39.999 | 20 000 |
| | Hollow shapes | All | 20 000 |
| ZK60A-T5 | Bars, rods, and shapes | 1.999 and under | 30 000 |
| | | 2.000 to 2.999 | 28 000 |
| | | 3.000 to 4.999 | 25 000 |
| | Hollow shapes | All | 26 000 |

TABLE IV.—UNIT DEFORMATION VALUES.
(Revision of Table XI, B 107 – 56 T)

| Alloy and Condition | Yield Strength (0.2 per cent offset) min, psi | Unit Deformation, in. per in. of gage length |
|---|---|---|
| AZ31B | 20 000 | 0.0051 |
| | 16 000 | 0.0045 |
| AZ61A | 20 000 | 0.0051 |
| | 24 000 | 0.0057 |
| | 22 000 | 0.0054 |
| | 16 000 | 0.0045 |
| AZ80A-F | 28 000 | 0.0063 |
| | 27 000 | 0.0062 |
| AZ80A-T51 | 30 000 | 0.0066 |
| | 32 000 | 0.0069 |
| ZK60A-F | 31 000 | 0.0068 |
| | 28 000 | 0.0063 |
| ZK60A-T5 | 36 000 | 0.0075 |
| | 38 000 | 0.0078 |

For alloy AM100B change the maximum silicon requirement from the present "1.00" to read "1.0" per cent.

### Tentative Specification for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 56 T):

*Section 2.*—In Item (*3*) revise "Temper" to read "Condition."

*Section 3.*—Revise the title to read "Materials and Manufacture."

*Section 7.*—Revise "temper" to read "condition."

*Section 9.*—Revise the title to read "Number of Tests."

*New Section.*—Add a new Section 14 titled "Packing, Marking, and Shipping" to read as follows, renumbering subsequent sections accordingly.

14. (*a*) The material shall be packed in such a manner as to prevent damage in ordinary handling and transportation. The type of packing and gross weight of individual containers shall be left to the discretion of the supplier unless otherwise agreed upon. Packing methods and containers shall be so selected as to permit maximum utility of mechanical equipment in unloading and subsequent handling. Each package or container shall contain only one size or alloy when packed for shipment unless otherwise agreed upon.

(*b*) Each package or container shall be marked with the purchase order number, quantity, specification number, alloy, gross and net weights and name of manufacturer.

(*c*) Packages or containers shall be such as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

*Table I.*—For alloy M1A change the minimum manganese content from the present "1.20" to read "1.2" per cent. Add alloy AZ31C with the following composition:

| | |
|---|---|
| Magnesium, per cent | remainder |
| Aluminum, per cent | 2.4 to 3.6 |

| | |
|---|---|
| Manganese, min, per cent | 0.15[a] |
| Zinc, per cent | 0.50 to 1.5 |
| Silicon, max, per cent | 0.10 |
| Copper, max, per cent | 0.10 |
| Nickel, max, per cent | 0.03 |
| Other impurities, max, per cent | 0.30 |

[a] Manganese limit need not be met if iron is 0.005 per cent or less.

*Table II.*—Revise to read as shown in the accompanying Table II.

*Tables III and VI.*—Revise the heading to read "plus and minus" in place of the present "plus or minus."

*Tables VII and VIII.*—Add a footnote referenced to the first column to read "Intermediate dimensions shall be rounded off to the third decimal place in accordance with the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E 29)."

*Table X.*—Revise to read as shown in the accompanying Table III.

*Table XI.*—Revise to read as shown in the accompanying Table IV.

*Explanatory Note 1.*—Add the following to this note:

> *Alloy AZ31C.*—This alloy has the same characteristics as alloy AZ31B except that the impurity limits are greater. It is used for applications where the maximum in corrosion resistance is not necessary.

**Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 56 T):**

*New Section.*—Add a new Section 13 as indicated above for new Section 13 in Specification B 26, renumbering subsequent sections accordingly.

**Tentative Specification for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 56 T):**

This specification has been completely revised and is appended hereto in its revised form.[2]

**Tentative Specification for Aluminum-Alloy Sheet and Plate (B 209 – 56 T):**

This specification has been completely revised and is appended hereto in its revised form.[2]

**Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 56 T):**

This specification has been completely revised and is appended hereto in its revised form.[2]

**Tentative Specification for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 56 T):**

*Section 1.*—Delete Note 2.

*Section 2.*—Add a new Item (4) to read as follows, renumbering subsequent items accordingly: "Whether yield strength tests are required (Section 7)."

*Section 3.*—Delete the second sentence.

*Section 7.*—Revise to read as follows:

> 7. The bars, rods and wire shall be supplied in the alloy, temper and size specified in the contract or order and shall conform to the requirements as to tensile properties prescribed in Table II. Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 990A, 996A, and GR20A, yield strength is not important. For these alloys yield strength need not be determined unless specifically indicated in the contract or purchase order.

*Section 8 (a).*—Add the word "or" following the word "material" in the first sentence.

*Section 12.*—Revise to read "Variations from the specified dimensions for the class of material ordered shall not exceed the amounts prescribed in Tables III, IV, V, VI, VII, and VIII."

*New Section.*—Add a new Section 15 titled "Packing, Marking, and Shipping" to read as follows, renumbering subsequent sections accordingly:

> 15. (*a*) The material shall be packed in such a manner as to prevent damage in ordinary handling and transportation. The type of packing and gross weight of individual containers shall be left to the discretion of the supplier unless otherwise agreed upon. Each package or container shall contain only one size, alloy or temper of material when packed for shipment unless otherwise agreed upon.

## TABLE V.—TENSILE REQUIREMENTS

(Revision of Table II, Specification B 211 – 56 T)

| Alloy | Temper | Diameter or Thickness, in. | Tensile Strength, psi |  | Yield Strength, psi[a] | Elongation in 2 in. or 4X diameter, min, per cent[c] |
|---|---|---|---|---|---|---|
|  |  |  | Minimum | Maximum |  |  |
| 990A | O | 0.124 and under | 11 000 | 15 500 | .... | .. |
|  |  | 0.125 and over | 11 000 | 15 500 | 3 000 min[b] | 25 |
|  | H14 | 0.374 and under | 16 000 | .... | .... | .. |
|  | H18 | 0.374 and under | 22 000 | .... | .... | .. |
|  | H112 | All | 11 000 | .... | 3 000 min[b] | .. |
|  | F | All | [d] | .... | [d] | .. |
| 996A | O | 0.124 and under | 9 500 | .... | 2 500 | .. |
|  |  | 0.125 and over | 9 500 | .... | 2 500 | 25 |
|  | H14 | 0.374 and under | 12 000 | .... | 10 000 | .. |
|  | H18 | 0.374 and under | 16 000 | .... | 13 000 | .. |
| CG42A | O | 0.124 and under | .... | 35 000 | .... | .. |
|  |  | 0.125 to 8.000 | .... | 35 000 | .... | 16 |
|  | T4 | 0.124 and under | 62 000 | .... | .... | .. |
|  |  | 0.125 to 6.500[e] | 62 000 | .... | 40 000 min | 14 |
| CM41A | O | 0.124 and under | .... | 35 000 | .... | .. |
|  |  | 0.125 to 8.000 | .... | 35 000 | .... | 16 |
|  | T4 | 0.124 and under | 55 000 | .... | .... | .. |
|  |  | 0.125 to 8.000[f] | 55 000 | .... | 32 000 min | 16 |
| CB60A | T3 | 0.125 to 1.500 | 45 000 | .... | 38 000 min | 10 |
|  |  | 1.501 to 2.000 | 43 000 | .... | 34 000 min | 12 |
|  |  | 2.001 to 3.000 | 42 000 | .... | 30 000 min | 14 |
|  | T8 | 0.125 to 3.250 | 52 000 | .... | 40 000 min | 10 |
| CS41A | O | 0.124 and under | .... | 35 000 | .... | .. |
|  |  | 0.125 to 8.000 | .... | 35 000 | .... | 12 |
|  | T4 | 0.124 and under | 55 000 | .... | .... | .. |
|  |  | 0.125 to 6.750[e] | 55 000 | .... | 32 000 min | 16 |
|  | T6 | 0.124 and under | 65 000 | .... | .... | .. |
|  |  | 0.125 to 6.750[e] | 65 000 | .... | 55 000 min | 8 |
| GR20A | O | 0.124 and under | .... | 32 000 | .... | .. |
|  |  | 0.125 and over | 25 000 | 32 000 | 9 500 min[b] | 25 |
|  | H34 | 0.374 and under | 34 000 | .... | 26 000 min[b] | .. |
|  | H38 | 0.374 and under | 39 000 | .... | .... | .. |
|  | F | All | [d] | [d] | .... | .. |
| GS11A | O | 0.124 and under | .... | 22 000 | .... | .. |
|  |  | 0.125 to 8.000 | .... | 22 000 | .... | 18 |
|  | T4 | 0.124 and under | 30 000 | .... | .... | .. |
|  |  | 0.125 to 8.000[f] | 30 000 | .... | 16 000 min | 18 |
|  | T6 | 0.124 and under[f] | 42 000 | .... | .... | .. |
|  |  | 0.125 to 8.000 | 42 000 | .... | 35 000 min | 10 |
| ZG62A | O | 0.124 and under | .... | 40 000 | .... | .. |
|  |  | 0.125 to 8.000 | .... | 40 000 | .... | 10 |
|  | T6 | 0.124 and under | 77 000 | .... | .... | .. |
|  |  | 0.125 to 4.000[g] | 77 000 | .... | 66 000 min | 7 |

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[a] Yield strength is defined as the stress which produces a permanent set of 0.2 per cent of the initial gage length.

[b] Yield strength to be determined only when specified in the contract or purchase order.

[c] The measurement of elongation is not required for wire less than 0.125 in. in thickness.

[d] There are no tensile requirements for material in the F temper but it usually can be expected that material 1½ in. or less in thickness or diameter (except sections over 4 in. in width) will have a strength about equivalent to the H14 or H34 temper. As size increases the strength decreases to nearly that of the 0 temper.

[e] For rounds, maximum diameter is 6.500 in.; for square, rectangular, hexagonal or octagonal bar, maximum thickness is 4 in. and 36 sq in. maximum cross-sectional area.

[f] For bar maximum cross-sectional area is 50 sq in.

[g] For rounds, maximum diameter is 4 in.; for square bar maximum size is 3½ by 3½ in.; for rectangular bar, maximum thickness is 3 in. with corresponding maximum width of 6 in. For rectangular bar less than 3 in. in thickness, maximum width is 10 in.

(*b*) Each package or container shall be marked with the purchase order number, size of material, specification number, alloy and temper, the latter to be in accordance with ASTM Codification Systems (ASTM Designations B 275 and B 296) or the commercial system, gross and net weights, and name of the manufacturer.

(*c*) Packages or containers shall be such as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

*Table I.*—Delete footnote *a*, relettering subsequent footnotes accordingly. Add chemical requirements for alloy 996A as follows:

| | |
|---|---|
| Aluminum, min, per cent | 99.60 |
| Copper, per cent | 0.05 |
| Iron, per cent | 0.35 |
| Silicon, per cent | 0.25 |
| Manganese, per cent | 0.03 |
| Magnesium, per cent | 0.03 |
| Zinc, per cent | 0.05 |
| Titanium, per cent | 0.03 |
| Other elements, per cent: | |
| Each | 0.03 |

*Table II.*—Revise to read as shown in the accompanying Table V.

*New Tables.*—Add new Tables VI, VII, and VIII to read as shown in the accompanying Tables VI, VII, and VIII.

*Appendix.*—Add to the table cross indexing ASTM and AA designation ASTM alloy 996A with the AA designation 1060.

### Tentative Specification for Magnesium-Base Alloy Extruded Tubes (B 217 - 56 T):

*Section 3.*—Revise the title to read "Materials and Manufacture."

*New Section.*—Add a new Section 14 titled "Packing, Marking, and Shipping" to read the same as indicated above for New Section 14 in Specification B 107, renumbering subsequent sections accordingly.

*Table I.*—Add alloy AZ31C with the following chemical requirements:

| | |
|---|---|
| Magnesium, per cent | remainder |
| Aluminum, per cent | 2.4 to 3.6 |
| Manganese, min, per cent | 0.15[a] |
| Zinc, per cent | 0.50 to 1.5 |
| Silicon, max, per cent | 0.10 |
| Copper, max, per cent | 0.10 |
| Nickel, max, per cent | 0.03 |
| Other impurities, max, per cent | 0.30 |

[a] Manganese limit need not be met if iron is 0.005 per cent or less.

*Tables V, VI, VII, VIII, IX, and XI.*—Add a footnote referenced after the heading for column 1 to read "Intermediate dimensions shall be rounded off to the third decimal place in accordance with the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E 29).[3]

*Tables III, IV, V, and VI.*—Revise the expression "plus or minus" to read "plus and minus."

*Table II.*—Revise the heading for column 1 from the present "Alloy and Temper" to read "Alloy and Condition."

### Tentative Specification for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 56 T):

*Section 2.*—Add a new Item (*4*) to read as follows, renumbering subsequent items accordingly "Whether yield strength tests are required (Section 7)."

TABLE VI.—PERMISSIBLE VARIATIONS FROM SPECIFIED SPECIFIC OR MULTIPLE LENGTH OF ROLLED OR COLD FINISHED RODS, BAR AND WIRE.

(New Table VI, Specification B 211 - 56 T)

| Diameter, Thickness or Distance Across Flats, in. | Permissible Variation in Length, plus, in. | | | |
|---|---|---|---|---|
| | Specified Length, ft | | | |
| | 12 and under | Over 12 to 30 | Over 30 to 50 | Over 50 |
| 2.999 and under | 1/8 | 1/4 | 3/8 | 1 |
| 3.000 to 7.999 | 3/16 | 5/16 | 7/16 | 1 |
| 8.000 and over | 1/4 | 3/8 | 1/2 | 1 |

*Section 7.*—Revise to read as follows:

7. The extruded bars, rods and shapes shall be supplied in the alloy, temper and size specified in the contract or order and shall conform to the requirements as to tensile properties prescribed in Table II. Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 990A, GR40A, and M1A, yield strength is not important. For these alloys yield strength

need not be determined unless specifically indicated in the contract or purchase order.

TABLE VII.—PERMISSIBLE VARIATION FROM STRAIGHTNESS OF ROLLED OR COLD FINISHED ROD AND BAR.

(New Table VII, Specification B 211 - 56 T)

| Product | Permissible Variation, in. | |
|---|---|---|
| | Up through 5 ft | Over 5 ft |
| Rolled | 0.250 | 0.050 × length in feet |
| Drawn or Cold Finished | 0.125 | 0.025 × length in feet |

TABLE VIII.—PERMISSIBLE VARIATION FROM SQUARENESS OF CUT ENDS OF ROLLED OR COLD FINISHED RODS AND BAR.

(New Table VIII, Specification B 211 - 56 T)

| Size | Permissible Variation, deg |
|---|---|
| All | 1 |

*Section 12.*—Revise to read as follows:

12. Variations from the specified dimensions for the class or type of materials ordered shall not exceed the amounts specified in Tables III, IV, V, VI, VII, VIII, IX, X, and XI.

*New Section.*—Add a new Section 15 titled "Packing, Marking, and Shipping" to read as indicated above for new Section 15 in Specification B 211, renumbering subsequent sections accordingly.

*Table I.*—Delete footnote *a*, relettering subsequent footnotes accordingly. Add chemical requirements for alloy GM51A as follows:

| | |
|---|---|
| Aluminum, per cent | remainder |
| Copper, per cent | 0.05 to 0.20 |
| Iron, per cent | [a] |
| Silicon, per cent | [a] |
| Manganese, per cent | 0.50 to 1.0 |
| Magnesium, per cent | 4.7 to 5.5 |
| Zinc, per cent | 0.25 |
| Chromium, per cent | 0.05 to 0.20 |
| Titanium, per cent | 0.10 |
| Beryllium, per cent | 0.0005 |
| Others, per cent: | |
| Each | 0.05 |
| Total | 0.15 |

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[a] The permissible maximum amount of iron plus silicon is 0.40 per cent.

*Table II.*—Revise to read as shown in the accompanying Table IX.

*New Tables.*—Add new Tables VI, VII, VIII, IX, X, and XI as shown in the accompanying Tables X, XI, XII, XIII, XIV, and XV.

*Appendix.*—Add to the table of cross index of ASTM and AA designations ASTM alloy GM51A with the AA designation 5456.

### Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 56 T):

*Section 2.*—Add a new Item (5) to read as follows, renumbering subsequent items accordingly: "Whether inside or outside cladding is required (Section 14, type M1A tubes only)."

*Section 13.*—Revise the first sentence to read "The tubing shall be capable of being flattened along any element under a gradually applied load until the minimum outside diameter is not more than eight times the wall thickness."

*Section 14 (a).*—Revise to read "The aluminum alloy coating of type M1A tubes shall comprise either the inside surface (only) and its thickness shall be approximately 10 per cent of the total wall thickness, or the outside surface (only) in which case its thickness shall be approximately 7 per cent of the total wall thickness."

*New Section.*—Add a new Section 18 titled "Packing, Marking, and Shipping" to read as indicated above for new Section 15 in Tentative Specification B 211, renumbering subsequent sections accordingly.

*Table I.*—Add chemical requirements for alloy 996A as indicated above for Table I in Specifications B 211.

Delete footnote sign "a" from M1A and M1A clad and add footnote sign "a" after word "alloy" (first column heading).

*Table II.*—For alloys GS11A and GS11C delete the listings for wall thicknesses of 0.260 to 0.500 in. For alloys GS11A, GS11C, M1A, and clad M1A revise the present indicated wall thickness to read "0.200" in. Add requirements for alloy 996A and revise the requirements for clad M1A to read as shown in the accompanying Table XVI.

*Table III.*—In column one revise the listing of specified diameter from the present "1.001 to 2.000, incl" to read "1.001 to 1.500, incl" in.

*Table VI.*—In column one revise the listing of specified diameter from the present "0.375 to 5.999, incl" to read "0.375 to 1.500, incl" in.

*Appendix.*—Add to the table of cross index of ASTM and AA designations ASTM alloy 996A with the AA designation 1060.

**Tentative Specification for Aluminum-Alloy Extruded Tubes (B 235 – 56 T):**

This specification has been completely revised and is appended hereto in its revised form.[2]

TABLE IX.—TENSILE REQUIREMENTS.
(Revision of Table II, Specification B 221 – 56 T)

| Alloy | Temper | Thickness, in.$^{a}$ | Area, sq in. | Tensile Strength, psi | | Yield Strength, min, psi$^{b}$ | Elongation in 2 in. or 4× diameter, min, per cent$^{c}$ |
|---|---|---|---|---|---|---|---|
| | | | | Minimum | Maximum | | |
| 990A | O | All | .... | 11 000 | 15 500 | 3 000$^{g}$ | 25 |
| | H112 | All | .... | 11 000 | .... | 3 000$^{g}$ | .. |
| | F | All | .... | $^{h}$ | .... | $^{h}$ | .. |
| CG42A | O | All | All | .... | 35 000 | 19 000 max | 12 |
| | T4$^{d}$ | 0.050 to 0.249 | All | 57 000 | .... | 42 000 | 12 |
| | | 0.250 to 0.749 | All | 60 000 | .... | 44 000 | 12 |
| | | 0.750 to 1.499 | All | 65 000 | .... | 46 000 | 10 |
| | | 1.500 and over | 25 and under | 70 000 | .... | 52 000 | 10 |
| | | 1.500 and over | Over 25 to 32 | 68 000 | .... | 48 000 | 8 |
| CS41A | O | All | All | .... | 30 000 | 18 000 max | 12 |
| | T4$^{e}$ | All | All | 50 000 | .... | 35 000 | 12 |
| | T6$^{f}$ | 0.125 to 0.499 | All | 60 000 | .... | 53 000 | 7 |
| | | 0.500 to 0.749 | All | 64 000 | .... | 58 000 | 7 |
| | | 0.750 and over | 25 and under | 68 000 | .... | 60 000 | 7 |
| | | 0.750 and over | Over 25 to 32 | 68 000 | .... | 58 000 | 6 |
| GM51A | O | 5.000 and under | 32 and under | 42 000 | .... | 19 000$^{g}$ | 16 |
| | H112 | 5.000 and under | 32 and under | 42 000 | .... | 19 000$^{g}$ | 12 |
| | H311 | 5.000 and under | 32 and under | 42 000 | .... | 25 000$^{g}$ | 12 |
| CR40A | O | All | .... | 30 000 | 41 000 | 11 000$^{g}$ | .. |
| | H112 | All | .... | 30 000 | .... | 11 000$^{g}$ | .. |
| GS10A | T4 | 0.500 and under | All | 22 000 | .... | 10 000 | 14 |
| | T42 | 0.500 and under | All | 17 000 | .... | 10 000 | 12 |
| | T5 | 0.500 and under | All | 22 000 | .... | 16 000 | 8 |
| | T6 | 0.124 and under | All | 30 000 | .... | 25 000 | 8 |
| | | 0.125 to 0.500 | All | 30 000 | .... | 25 000 | 10 |
| GS11A | O | All | All | .... | 22 000 | 16 000 max | 16 |
| | T4 | All | All | 26 000 | .... | 16 000 | 16 |
| | T6 | All | All | 38 000 | .... | 35 000 | 10 |
| | T62 | All | All | 35 000 | .... | 26 000$^{i}$ | 10 |

TABLE IX.—*Continued.*

| Alloy | Temper | Thickness, in.[a] | Area, sq. in. | Tensile Strength, psi | | Yield Strength, min, psi[b] | Elongation in 2 in. or 4X diameter, min per cent |
|---|---|---|---|---|---|---|---|
| | | | | Minimum | Maximum | | |
| GS11C | O | All | All | .... | 22 000 | 16 000 max | 16 |
| | T4 | All | All | 26 000 | .... | 16 000 | 16 |
| | T6 | All | All | 38 000 | .... | 35 000 | 10 |
| M1A | O | All | All | 14 000 | 19 000 | 5 000[g] | .. |
| | H112 | All | All | 14 000 | .... | 5 000[g] | .. |
| | F | All | All | [i] | .... | [h] | .. |
| ZG62A | O | All | All | .... | 40 000 | 24 000 max | 10 |
| | T6 | 0.249 and under | All | 78 000 | .... | 70 000 | 7 |
| | | 0.250 to 2.999 | All | 80 000 | .... | 72 000 | 7 |
| | | 3.000 to 4.499 | 20 and under | 80 000 | .... | 70 000 | 7 |
| | | 3.000 to 4.499 | Over 20 to 32 | 78 000 | .... | 70 000 | 6 |
| | | 4.500 to 5.000 | 32 and under | 78 000 | .... | 68 000 | 6 |

[a] Specimens tested parallel to direction of extrusion.

[b] Yield strength is defined as the stress which produces a permanent set of 0.2 per cent of the initial gage length.

[c] For material for which a standard test specimen cannot be taken, or for material thinner than 0.062 in., the test for elongation is not required.

[d] CG42A material heat treated by the purchaser regardless of thickness shall develop a minimum tensile strength of 57,000 psi, a minimum yield strength of 38,000 psi, and a minimum elongation of 12 per cent for thickness 0.749 in. and under, 10 per cent for thicknesses 0.750 and over, except that if the cross-sectional area is over 25 sq in. the elongation shall be 8 per cent min. The temper of such material is T42.

[e] CS41A material heat treated by the purchaser shall develop a minimum yield strength of 29,000 psi and is properly designated as T42 temper.

[f] CS41A material heat treated and aged by the purchaser regardless of thickness shall develop a minimum tensile strength of 60,000 psi, a minimum yield strength of 53,000 psi, and a minimum elongation of 7 per cent, except that if the cross-sectional area is over 25 sq in. the minimum elongation shall be 6 per cent. The temper of such material is T62.

[g] Yield strength shall be determined only when specified in the contract or purchase order.

[h] There are no tensile requirements for alloys 990A and M1A in the F temper but the tensile strength of alloy 990A in the F temper (as extruded) is about 13,000 to 17,000 psi and of alloy M1A about 16,000 to 21,000 psi, the lower values being associated with thick or bulky sections.

[i] Yield strength not to exceed 80 per cent of tensile strength.

## Tentative Specification for Aluminum Bars for Electrical Purposes (Bus Bars (B 236 – 56 T):

*New Section.*—Add a new Section 19 titled "Packing, Marking, and Shipping" to read as follows, renumbering subsequent sections accordingly:

19. (*a*) The material shall be properly and adequately bundled, crated, or otherwise packaged to protect it against injury in ordinary handling and transportation. The type of packing shall be left to the discretion of the manufacturer, unless otherwise agreed upon. Each package or container shall contain only one size, type, or temper of material when packaged for shipment unless otherwise agreed upon.

(*b*) Each package or container shall be marked with the purchase order number, size, quantity, specification number, type and temper, gross and net weights, and name of manufacturer.

(*c*) Packages or containers shall be such as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

## Tentative Specification for Aluminum-Alloy Pipe (B 241 – 56 T):

*Section 1.*—Revise to read "This specification covers schedules 5, 10, 40 (standard) and 80 (extra heavy) pipe, either drawn or extruded, of the aluminum alloys designated in Table I."

### TABLE X. PERMISSIBLE VARIATION IN ANGULARITY.

(New Table VI, Specification B 221 - 56 T)

| Specified Leg Thickness, in. | Permissible Variation from Specified or Nominal Angle, deg | |
|---|---|---|
| | Ratio[a,b] Leg or Surface Length to Leg or Metal Thickness | |
| | 1 and under | Over 1 |
| Col. 1 | Col. 2 | Col. 3 |
| 0.187 and under | 1 | 2 |
| 0.188 to 0.749 | 1 | 1½ |
| 0.750 and over | 1 | 1 |

[a] When the space between the surfaces forming an angle is all metal, values in column 2 apply if the larger surface length to metal thickness ratio is one or less.

[b] When two legs are involved, the one having the larger ratio determines the applicable column.

### TABLE XI.—PERMISSIBLE VARIATION FROM SPECIFIED LENGTH.

(New Table VII, Specification B 221 - 56 T)

| Circumscribing Circle Diameter (Shapes);[a] Specified Diameter (Rod); Specified Width or Depth Whichever Greater (Bar), in. | Permissible Variation in Length, plus, in.[b] | | | |
|---|---|---|---|---|
| | Specified Length, ft | | | |
| | 12 and under | Over 12 to 30 | Over 30 to 50 | Over 50 |
| 2.999 and under | ⅛ | ¼ | ⅜ | 1 |
| 3.000 to 7.999 | 3⁄16 | 5⁄16 | 7⁄16 | 1 |
| 8.000 and over | ¼ | ⅜ | ½ | 1 |

[a] The circumscribing circle diameter is the diameter of the smallest circle that will completely enclose the shape.

[b] Applicable only to straight lengths.

### TABLE XII.—PERMISSIBLE VARIATION FROM FLATNESS (FLAT SURFACES).

(New Table VIII, Specification B 221 - 56 T)

| Surface Width | Permissible Deviation, in.[a] | |
|---|---|---|
| | Minimum | Maximum |
| All | 0.004 | 0.004 × surface width in inches |

[a] Not applicable to surface of walls 0.250 in. or less in thickness which completely enclose a void.

*Section 2.*—Add a new Item (*4*) to read as follows, renumbering subsequent items accordingly; "Whether yield strength tests are required (Section 7)."

*Section 7.*—Revise to read as follows:

7. Pipe shall be supplied in the alloy and temper specified in the contract or purchase order and shall conform to the requirements as to tensile properties prescribed in Table II. Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloy M1A, yield strength is not important. For this alloy, yield strength need not be determined unless specifically indicated in the contract or purchase order.

*Section 12.*—Revise to read as follows:

12. (*a*) *Diameter.*—Diameter of pipe shall not vary from the nominal diameter as given in Table III by an amount greater than the values shown in Table IV.

(*b*) *Wall Thickness.*—The wall thickness shall not vary from the nominal wall thickness

### TABLE XIII.—PERMISSIBLE VARIATION FROM SPECIFIED CONTOUR (CURVED SURFACES).

(New Table IX, Specification B 221 - 56 T)

Permissible Deviation: 0.005 in. per in. of Chord Length; 0.005 in. min (Applicable to not more than 90 deg of any arc)

### TABLE XIV.—PERMISSIBLE DEPTH OF SURFACE DEFECTS.

(New Table X, Specification B 221 - 56 T)

| Specified Thickness, in. | Permissible Depth of Defect, max, in.[a] |
|---|---|
| 0.063 and under | 0.0015 |
| 0.064 to 0.125 | 0.002 |
| 0.126 to 0.188 | 0.0025 |
| 0.189 to 0.250 | 0.003 |
| 0.251 to 0.500 | 0.004 |
| 0.501 and over | 0.008 |

[a] Defects include extrusion die lines and handling marks.

### TABLE XV.—PERMISSIBLE VARIATION FROM SQUARENESS OF CUT ENDS.

(New Table XI, Specification B 221 - 56 T)

| Size | Permissible Variation, deg |
|---|---|
| All | 1 |

TABLE XVI.—TENSILE REQUIREMENTS.
(Partial Revision of Table II, Specification B 234 - 56 T)

| Alloy | Temper | Wall Thickness, in. | Tensile Strength, min, psi | Yield Strength, min, psi | Elongation in 2 in. or 4× diameter, min, per cent | |
|---|---|---|---|---|---|---|
| | | | | | Full-Section Specimens | Cut-Out Specimens |
| 996A | H14 | 0.010 to 0.500 | 12 000 | 10 000 | .... | .... |
| Clad M1A | H14 | 0.010 to 0.500 | 19 000 | 16 000 | .... | .... |

TABLE XVII.—TENSILE REQUIREMENTS.[a]
(Revision of Table II, Specification B 241 - 56 T)

| Alloy[b] | Temper | Pipe Size, in. | Tensile Strength, min, psi | Yield Strength, min, psi[c] | Elongation in 2 in. or 4 × diameter, min. per cent |
|---|---|---|---|---|---|
| GS10A | T5 | All | 22 000 | 16 000 | 10 |
| | T6 | All | 30 000 | 25 000 | 8 |
| GS11A | T6 | Under 1 | 42 000 | 35 000 | 12 |
| | | 1 and Over | 38 000 | 35 000 | 10 |
| GS11C | T6 | Under 1 | 42 000 | 35 000 | 12 |
| | | 1 and Over | 38 000 | 35 000 | 10 |
| M1A | H18 | Under 1 | 27 000 | 24 000[d] | .. |
| | H112 | 1 and Over | 14 500 | 6 000[d] | .. |

[a] Specimens tested parallel to the direction of working.

[b] These alloy designations were established in accordance with the Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (ASTM Designation: B 275).[3]

[c] Yield strength is defined as the stress which produces a permanent set of 0.2 per cent of the initial gage length.

[d] Yield strength to be determined only when specified in the contract or purchase order.

as given in Table III by an amount greater than the values shown in Table V.

(c) *Length*.—The length of each piece of pipe shall not be less than that specified and shall not exceed the specified amount by more than ¼ in. For specified lengths over 20 ft the allowable variation from specified length shall be negotiated between the manufacturer and the purchaser.

(d) *Weight*.—The weight of the pipe shall not vary from the theoretical weight as given in Table III by an amount greater than the values shown in Table VI.

(e) *Straightness*.—The pipe shall not vary from straight by an amount greater than the values shown in Table VII.

*Section 13 (a)*. Delete the last sentence.

*New Section*.—Add a new Section 15 titled "Packing, Marking, and Shipping" to read as follows, renumbering subsequent sections accordingly:

15. (*a*) The material shall be packed in such a manner as to prevent damage in ordinary handling and transportation. The type of packing and gross weight of individual containers shall be left to the discretion of the supplier unless otherwise agreed upon. Each package or container shall contain only one size, alloy, temper or schedule number of material when packed for shipment unless otherwise agreed upon.

(*b*) Each package or container shall be marked with the purchase order number, size and schedule number of material, specification number, alloy and temper, the latter to be in accordance with ASTM Codification Systems (ASTM Designations B 275 and B 296) or the commercial system, gross and net weights, and name of manufacturer.

(*c*) Packages or containers shall be such as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

TABLE XVIII. NOMINAL DIMENSIONS AND WEIGHTS OF ALUMINUM-ALLOY PIPE.
(Revision of Table III, Specification B 241 – 56 T)

| Size, in. | Outside Diameter, in. | ASA Schedule 5 | | ASA Schedule 10 | | Standard Pipe (ASA Schedule 40) | | Extra Heavy Pipe (ASA Schedule 80) | |
|---|---|---|---|---|---|---|---|---|---|
| | | Wall Thickness, in. | Weight, lb per ft[a] | Wall Thickness, in. | Weight, lb per ft[a] | Wall Thickness, in. | Weight, lb per ft[a] | Wall Thickness, in. | Weight, lb per ft[a] |
| ⅛ | 0.405 | ..... | ..... | 0.049 | 0.064 | 0.068 | 0.085 | 0.095 | 0.109 |
| ¼ | 0.540 | ..... | ..... | 0.065 | 0.114 | 0.088 | 0.147 | 0.119 | 0.185 |
| ⅜ | 0.675 | ..... | ..... | 0.065 | 0.146 | 0.091 | 0.196 | 0.126 | 0.256 |
| ½ | 0.840 | 0.065 | 0.186 | 0.083 | 0.232 | 0.109 | 0.294 | 0.147 | 0.376 |
| ¾ | 1.050 | 0.065 | 0.237 | 0.083 | 0.297 | 0.113 | 0.391 | 0.154 | 0.510 |
| 1 | 1.315 | 0.065 | 0.300 | 0.109 | 0.486 | 0.133 | 0.581 | 0.179 | 0.751 |
| 1¼ | 1.660 | 0.065 | 0.383 | 0.109 | 0.625 | 0.140 | 0.786 | 0.191 | 1.037 |
| 1½ | 1.900 | 0.065 | 0.441 | 0.109 | 0.721 | 0.145 | 0.940 | 0.200 | 1.256 |
| 2 | 2.375 | 0.065 | 0.555 | 0.109 | 0.913 | 0.154 | 1.264 | 0.218 | 1.737 |
| 2½ | 2.875 | 0.083 | 0.856 | 0.120 | 1.221 | 0.203 | 2.004 | 0.276 | 2.650 |
| 3 | 3.500 | 0.083 | 1.048 | 0.120 | 1.498 | 0.216 | 2.621 | 0.300 | 3.547 |
| 3½ | 4.000 | 0.083 | 1.201 | 0.120 | 1.720 | 0.226 | 3.151 | 0.318 | 4.326 |
| 4 | 4.500 | 0.083 | 1.354 | 0.120 | 1.942 | 0.237 | 3.733 | 0.337 | 5.183 |
| 5 | 5.563 | 0.109 | 2.196 | 0.134 | 2.688 | 0.258 | 5.057 | 0.375 | 7.188 |
| 6 | 6.625 | 0.109 | 2.624 | 0.134 | 3.213 | 0.280 | 6.564 | 0.432 | 9.884 |
| 8 | 8.625 | 0.109 | 3.429 | 0.148 | 4.635 | 0.322 | 9.878 | 0.500 | 15.01 |
| 8 | 8.625 | ..... | ..... | ..... | ..... | $0.277^b$ | 8.543 | ...... | ...... |
| 10 | 10.750 | 0.134 | 5.256 | 0.165 | 6.453 | 0.365 | 14.00 | 0.593 | 23.25 |
| 10 | 10.750 | ..... | ..... | ..... | ..... | $0.307^b$ | 11.84 | ...... | ...... |
| 10 | 10.750 | ..... | ..... | ..... | ..... | $0.279^c$ | 10.79 | $0.500^d$ | 18.93 |
| 12 | 12.750 | 0.165 | 7.672 | 0.180 | 8.359 | 0.406 | 18.52 | 0.687 | 30.62 |
| 12 | 12.750 | ..... | ..... | ..... | ..... | $0.330^b$ | 15.14 | $0.500^d$ | 22.63 |

[a] The weights are based on nominal dimensions, plain ends, and a density of 0.098 lb per cu in. For alloy M1A multiply weights given in the table by 1.01.

[b] Schedule 30.

[c] No ASA Schedule.

[d] Schedule 60.

*Table II.*—Revise to read as shown in the accompanying Table XVII.

*Table III.*—Revise to read as shown in the accompanying Table XVIII.

*New Tables.*—Add new Tables IV, V, VI, and VII as shown in the accompanying Tables XIX, XX, XXI, and XXII.

## Tentative Specification for Aluminum-Alloy Die Forgings (B 247 – 55 T):

*New Section.*—Add a new Section 15 titled "Packing, Marking, and Shipping" to read as indicated above for new Section 15 in Specification B 211, renumbering subsequent sections accordingly.

*Table II.*—Add a new footnote *e* to read "Not applicable for forgings more than 4 in. in thickness." This footnote is to be referenced after the tensile strength, yield strength, elongation, and Brinell hardness requirements for alloys CN42C, CN42D, CS41A, CS41C, GS11A, GS11B, SG11A, and SG121A.

## Tentative Specification for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 – 56 T):

*New Section.*—Add a new Section 18 titled "Packing, Marking, and Shipping" to read as indicated above for new Section 15 in Specification B 211, renumbering subsequent sections accordingly.

## Tentative Specification for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 – 56 T):

*New Section.*—Add a new Section 14

titled "Packing, Marking, and Shipping" to read as indicated above for new Section 15 in Specification B 211, renumbering subsequent sections accordingly.

TABLE XIX.—PERMISSIBLE VARIATIONS IN OUTSIDE DIAMETER.

New Table IV, Specification B 241 – 56 T)

| Pipe Size, in. | Permissible Variations in Outside Diameter from the Nominal Diameter, in.[a] | |
|---|---|---|
| | Schedule 5 and 10 | Schedule 20 and Greater |
| Under 2........ | +0.015, −0.031 | +0.015, −0.031 |
| 2 to 4.......... | +0.031, −0.031 | +1 per cent, −1 per cent |
| 4½ to 7........ | +0.062, −0.031 | +1 per cent, −1 per cent |
| 8 to 12......... | +0.093, −0.031 | + 1 per cent, −1 per cent |

[a] Nominal diameters are as shown in Table III.

TABLE XX.—PERMISSIBLE VARIATIONS IN WALL THICKNESS.

(New Table V, Specification B 241 – 56 T)

| Pipe Schedule No. | Permissible Variations in Wall Thickness from the Nominal Wall Thickness, per cent[a] |
|---|---|
| 5 and 10................ | ±12½ |
| 20 and greater........... | −12½[b] |

[a] Nominal wall thicknesses are those shown in Table III.

[b] Maximum wall thickness is controlled by weight tolerance.

*Table I.*—Add chemical requirements for alloy GM51A as indicated above for Table I in Specification B 221 and for alloy GS11C indicating the same composition limits as now shown for alloy GS11A except chromium limits to be 0.04 to 0.14 per cent.

*Table II.*—Add tensile requirements

TABLE XXI.—PERMISSIBLE VARIATIONS IN WEIGHT.

(New Table VI, Specification B 241 – 56 T)

| Pipe Schedule No. | Permissible Variations in Weight from the Nominal Weight[a] |
|---|---|
| 5 and 10................ | [b] |
| 20 and greater........... | +8 per cent[c] |

[a] Nominal weight is that shown in Table III for the specified size and schedule no.

[b] For schedules 5 and 10, only diameter, wall thickness and length tolerances apply.

[c] Minimum weight is controlled by tolerances for outside diameter and wall thickness.

TABLE XXII.—PERMISSIBLE VARIATIONS FROM STRAIGHTNESS.

(New Table VII, Specification B 241 – 56T)

| Pipe Size, in. | Permissible Variation, in. | |
|---|---|---|
| | In Each Foot of Length | In Total Length of Piece |
| Under 6......... | 0.010 | 0.010 × length in feet |
| 6 to 12.......... | 0.020 | 0.020 × length in feet |

TABLE XXIII. TENSILE REQUIREMENTS.

(Addition to Table II, Specification B 308 – 56 T)

| Alloy | Temper | Thickness, in. | Area, sq in. | Tensile Strength, min, psi | Yield Strength, min, psi | Elongation in 2 in. or 4× Diameter, min. per cent |
|---|---|---|---|---|---|---|
| GM51A...... | O......... | 5.000 and under | 32 and under | 42 000 | 19 000 | 16 |
| | H112...... | 5.000 and under | 32 and under | 42 000 | 19 000 | 12 |
| | H311...... | 5.000 and under | 32 and under | 42 000 | 25 000 | 12 |
| GS11A........ | T62....... | ... | ... | 35 000 | 26 000[a] | 10 |
| GS11C....... | T4........ | ... | ... | 26 000 | 16 000 | 16 |
| | T6........ | ... | ... | 38 000 | 35 000 | 10 |

[a] Yield strength not to exceed 80 per cent of the tensile strength.

for alloys GS11A, GS11C, and GM51A as shown in the accompanying Table XXIII.

*Appendix.*—Add to the table of cross index of ASTM and AA designations ASTM alloys GM51A and GS11C with corresponding AA designations 5456 and 6062, respectively.

TABLE XXIV.—CHEMICAL REQUIREMENTS.

(Revision of impurity requirements in Table I, Specification B 37 – 49)

| Grade | Copper, max, per cent | Zinc, max, per cent | Magnesium, max, per cent | Total of All Impurities, max, per cent |
|---|---|---|---|---|
| 980A...... | 0.2 | 0.2 | 0.5 | 2.0 |
| 950A...... | 1.5 | 1.5 | 1.0 | 5.0 |
| 920A...... | 4.0 | 1.5 | 1.0 | 8.0 |
| 900A...... | 4.5 | 3.0 | 2.0 | 10.0 |
| 850A...... | 5.0 | 5.5 | 2.5 | 15.0 |

### Tentative Specification for Aluminum-Alloy Round Welded Tubes (B 313 – 56 T):

*New Section.*—Add a new Section 15 titled "Packing, Marking, and Shipping" to read as indicated above for new Section 15 in Specification B 211, renumbering subsequent sections accordingly.

## REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the following three standards be revised immediately as indicated and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

### Standard Specification for Aluminum for Use in Iron and Steel Manufacture (B 37 – 49):

*New Section.*—Add a new Section 8 to read as follows, renumbering subsequent sections accordingly.

8. (*a*) The material shall be packed in such a manner as to prevent damage in ordinary handling and transportation. The type of packing and gross weight of individual containers shall be left to the discretion of the supplier unless otherwise agreed upon. Packing methods and containers shall be so selected as to permit maximum utility of mechanical equipment in unloading and subsequent handling. Each package or container shall contain only one size or grade of material when packed for shipment unless otherwise agreed upon.

(*b*) Each package or container shall be marked with the purchase order number, quantity, specification number, grade, gross and net weights, and name of the manufacturer.

(*c*) Package or containers shall be such as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

*Table I.*—Revise the impurity requirements to read as shown in the accompanying Table XXIV.

### Standard Specification for Magnesium Ingot and Stick for Remelting (B 92 – 56):

*Section 1.*—Change the words "99.8 per cent magnesium" to read "99.80 per cent magnesium."

*Section 3.*—Revise the title to read "Materials and Manufacture." Letter the present text as Paragraph (*a*) and add a new Paragraph (*b*) to read "The materials used shall be such as to produce products that will conform to the requirements as to chemical composition prescribed in this specification."

*New Section.*—Add a new Section 5 titled "Sampling for Chemical Analysis" to read as follows, renumbering subsequent sections accordingly:

5. (*a*) The sample for chemical analysis shall be taken by sawing, drilling, or milling the sample ingot or stick in such a manner as to be representative of its average cross-section.

(*b*) The saw, drill, or cutter used for taking the sample shall be thoroughly cleaned. No lubricant shall be used in the operation and the sawings or metal chips shall be carefully treated with a magnet to remove any particles of iron introduced in taking the sample.

*New Section.*—Add a new Section 8 titled "Packing, Marking, and Shipping" to read as follows, renumbering subsequent sections accordingly.

8. (*a*) The material shall be packed in such a manner as to prevent damage in ordinary handling and transportation. The type of packing and gross weight of individual containers shall be left to the discretion of the supplier unless otherwise agreed upon. Packing methods and containers shall be so selected as to permit maximum utility of mechanical equipment in unloading and subsequent handling. Each package or container shall contain only one size and form of material when packed for shipment unless otherwise agreed upon.

(*b*) Each package or container shall be marked with the purchase order number, form, quantity, specification number, gross and net weights, and name of manufacturer.

(*c*) Packages or containers shall be such as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

*Section* 8.—Delete this section titled "Sampling for Umpire Analysis."

*Table I.*—Add Notes 1 and 2 as indicated above for Table I in Tentative Specification B 93.

### Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 56):

*New Section.*—Add a new Section 8 titled "Packing, Marking, and Shipping" to read as follows, renumbering subsequent sections accordingly.

8. (*a*) The material shall be packed in such a manner as to prevent damage in ordinary handling and transportation. The type of packing and gross weight of individual containers shall be left to the discretion of the supplier unless otherwise agreed upon. Packing methods and containers shall be so selected as to permit maximum utility of mechanical equipment in unloading and subsequent handling. Each package or container shall contain only one size or alloy of material when packed for shipment unless otherwise agreed upon.

(*b*) Each package or container shall be marked with the purchase order number, quantity, specification number, alloy, gross and net weights, and the name of the manufacturer.

(*c*) Packages or containers shall be such as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery.

*Table I.*—For alloy CS43A revise the maximum magnesium requirement from the present "0.05" to read "0.10" per cent.

## WITHDRAWAL OF TENTATIVES

The committee recommends withdrawal of the following tentatives:

### Tentative Specifications for Aluminum-Sheet and Plate for Pressure Vessel Applications (B 178 – 56 T):

The requirements of Specifications B 178 are recommended for incorporation in the 1957 revision of the Tentative Specification for Aluminum-Alloy Sheet and Plate (ASTM Designation: B 209), as appended hereto.[2]

### Tentative Specifications for Aluminum-Alloy Bars, Rods, and Shapes for Pressure Vessel Applications (B 273 – 56 T):

The requirements of Specifications B 273 are recommended for incorporation in the 1957 revisions of the following specifications, as covered earlier in this report:

*Tentative Specifications for:*

Aluminum-Alloy Bars, Rods, and Wire (ASTM Designation: B 211), requirements for bars, rods, and shapes, both rolled and cold-finished,

Aluminum-Alloy Extruded Bars, Rods, and Shapes (ASTM Designation: B 221), requirements for extruded bars, rods, and shapes, and

Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (ASTM Designation: B 308), requirements for standard structural shapes, rolled or extruded.

### Tentative Specifications for Aluminum-Alloy Pipe and Tube for Pressure Vessel Applications (B 274 – 56 T):

The requirements of Specifications B 274 are recommended for incorporation in the 1957 revisions of the following specifications two of which are appended hereto,[2] and the other covered earlier in this report:

*Tentative Specifications for:*

Aluminum-Alloy Drawn Seamless Tubes (ASTM Designation: B 210), requirements for drawn seamless tubes,
Aluminum-Alloy Extruded Tubes (ASTM Designation: B 235), requirements for extruded tubes, and
Aluminum-Alloy Pipe (ASTM Designation: B 241), requirements for pipe.

## REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

*Standard Method of Test for:*

Dielectric Strength of Anodically Coated Aluminum (B 110 - 45),
Sealing of Anodically Coated Aluminum (B 136 - 45), and
Weight of Coating on Anodically Coated Aluminum (B 137 - 45).

## EDITORIAL CHANGE

The committee recommends an editorial change in one test method as follows:

**Standard Method of Test for Sealing of Anodically Coated Aluminum (B 136 - 45):**

*Section 2.*—Change the words "Anthraquinone Violet R" to read "Anthraquinone Violet RN."

# REPORT OF COMMITTEE B-8

ON

# ELECTRODEPOSITED METALLIC COATINGS*

Committee B-8 on Electrodeposited Metallic Coatings held two regular meetings during the year: on October 19, 1956, and on February 8, 1957, at the Benjamin Franklin Hotel in Philadelphia, Pa., and one informal meeting on September 20, 1956, at Los Angeles, Calif.

The committee consists of 129 members, of whom 55 are classified as producers, 28 as consumers, and 56 as general interest. A paper by Messrs. Katz, Proctor, and Nagley on "Performance of Various Chromate Supplementary Films on Electrodeposited Zinc Coatings," was presented at the February meeting of Subcommittee V and is appended hereto.[1]

## NEW TENTATIVES

The committee recommends for publication as tentative the following recommended practice as appended hereto:[2]

*Recommended Practice for the Preparation of:* Lead and Lead Alloys for Electroplating.

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Papers, Specifications and Definitions* (Fielding Ogburn, chairman).—Section C, under the chairmanship of Walter Moline, is preparing a monograph on processes and coatings of Electroless Nickel.

The Symposium Committee, under the chairmanship of W. L. Pinner, sponsored a very successful Symposium on Electrodeposited Coatings, which was presented not only at the Buffalo meeting in February but at the Second Pacific Area Meeting of the Society. The Symposium has been issued as separate publication, *ASTM STP No. 197*.

A task group under the chairmanship of Edward Saubestre, will evaluate the idea of marking coatings to indicate compliance with ASTM specifications.

*Subcommittee II on Performance Tests* (W. L. Pinner, chairman).—Program-4 panels were placed on exposure at Kure Beach, N. C., on April 28, 1956, and at Detroit, Mich., on May 1, 1956. The panels at Kure Beach deteriorated rapidly and were inspected in November, 1956. Inspections will be made of panels at both exposure sites during May, 1957. Good agreement was obtained on ratings made with the Lowenheim and the Durbin methods.

Section F, J. W. Kerstetter, chairman, will study the preparation and corrosion performance of plated coatings on aluminum.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] See p. 203.

[2] The new recommended practice appears in the 1957 Supplement to Book of ASTM Standards, Part 2.

[3] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee III on Conformance Tests* (R. F. Ledford, chairman).—Section A, Arthur DuRose, chairman, is preparing a program for the evaluation of new methods used for determination of thickness of electrodeposits. The work of Section C, under the chairmanship of M. M. Beckwith, comprises determination of hardness of electrodeposits.

Section F, under the chairmanship of L. M. Morse, has outlined a program for determination of stress in electrodeposits.

*Subcommittee IV on Electroplating Practice* (M. Frager, chairman).—Section W, under the chairmanship of Henry Kafarski, will establish limits of impurities in waters used in electroplating, cleaning, and phosphate coating. A task group, under the chairmanship of B. Knapp, is establishing a recommended practice in regard to nickel-on-nickel coatings.

*Subcommittee V on Supplementary Treatments for Electrodeposited Metallic Coatings* (M. Darrin, chairman) approved the methods of applying organic coatings on chromium plate, prepared by Section F, under the chairmanship of F. L. Scott, and they will be submitted to letter ballot of the main committee.

This report has been submitted to letter ballot of the committee, which consists of 127 voting members; 115 members returned their ballots, of whom 99 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
*Chairman.*

D. M. BIGGE,
*Secretary.*

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EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-8 presented to the Society through the Administrative Committee on Standards a new Tentative Recommended Practice for Preparation of Iron Castings for Electroplating. The new tentative recommended practice was accepted by the Standards Committee on August 15, 1957, and it appears in the 1957 Supplement to Book of ASTM Standards, Part 2, bearing the designation B 320 - 57 T.

## APPENDIX

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# PERFORMANCE OF VARIOUS CHROMATE SUPPLEMENTARY FILMS ON ELECTRODEPOSITED ZINC COATINGS*

By H. L. Katz,[1] K. L. Proctor,[1] and F. Nagley[2]

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## Synopsis

Various accelerated testing procedures were investigated and correlated with service performance in marine environments and evaluated as standard methods for quality-control of supplementary chromate films on electrodeposited zinc coatings. Ten commercial chromate treatments, applied to three thicknesses of zinc coating, were employed in the test. The depletion rate of hexavalent chromium and contribution of trivalent chromium to the corrosion-inhibiting properties of the film were studied. Determination of hexavalent chromium content and resistance to salt spray were found suitable as quality-control methods. Spot tests were found to be unfeasible. Protection afforded by chromate treatments to electroplated zinc coatings of 0.0002, 0.0005, and 0.0010 in. thickness appeared to be similar. However, inasmuch as the protection provided by the zinc is affected by the treatment, a zinc coating thickness of 0.0002 in. appears inadequate for application of supplementary chromate treatments.

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Under common conditions of atmospheric exposure, zinc reacts with water, oxygen, and acidic substances, such as carbon dioxide, to form insoluble white corrosion products. These products are commonly named "white rust" and have been found to be primarily a basic zinc carbonate of variable composition. On galvanized steel, zinc is used as a sacrificial coating and affords protection to the steel. In the presence of an electrolyte, such as when galvanized surfaces are left in contact with a slowly drying stagnant film of water, "white rust" results. This type of corrosion can be objectionable in many ways. For example, the white deposit may interfere with moving parts. Since the white deposit results from the conversion of zinc to salts, continued attack may consume all the zinc and lead to early rusting of the underlying steel. To minimize and retard this corrosion, supplementary chromate treatments have been applied to galvanized steel.

Problems have arisen in standardizing test methods for measuring the protection afforded by different supplementary chromate films on zinc coatings. The better known methods include outdoor exposure tests, salt spray (fog) tests, and various humidity tests. Few of these, however, have proven adequate as quality control tests since they are neither sufficiently rapid nor reproducible to be used for control purposes. The need exists for the development and standardization of accelerated test procedures which have satisfactory speed and precision for evaluating the quality of supplementary chromate films.

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* Presented at a meeting of Subcommittee V on Supplementary Treatment for Electrodeposited Metallic Coatings, on February 8, 1957, at Philadelphia, Pa.

[1] Industrial Test Laboratory, Philadelphia Naval Shipyard, Philadelphia, Pa.

[2] Bureau of Ships, Department of the Navy, Washington 25, D. C.

The primary objective of this study was to investigate and standardize test methods for the quality control of chromate supplementary films applied to electrodeposited zinc coatings for use in marine environments. Efforts were directed toward: (*a*) conducting exposure tests in marine environments for evaluating the quality of various chromate films; (*b*) development and investigation of accelerated tests. Based on theoretical considerations, emphasis has been placed upon correlation of the benefits expected by the use of chemically chromated zinc coatings with the hexavalent and trivalent chromium content of the film.

## DESCRIPTION OF MATERIALS

### *Test Panels*:

More than two thousand 4 by 6 in. and 3 by 4 in. steel test panels were fabricated and electroplated with zinc by a commercial firm. The test panels were prepared from 18 gage cold-rolled SAE type 1020 steel and plated with nominal 0.0010-, 0.0005-, and 0.0002-in. zinc coating conforming to the requirements of Federal Specification QQ-Z-325.[3] The three thicknesses of zinc correspond to classes 1, 2, and 3, type 1, of the Federal Specification. The acceptable zinc coating thickness requirements as specified for this test were:

| | *Minimum* | *Maximum* |
|---|---|---|
| Class 1 | 0.0010 in. | 0.0013 in. |
| Class 2 | 0.0005 in. | 0.0007 in. |
| Class 3 | 0.0002 in. | 0.0003 in. |

All panels were individually checked for compliance to zinc thickness requirements by means of an Aminco-Brenner Magne-Gage.[4] It was noted that the deposit was somewhat heavier toward the edges.

### *Treatments*:

Ten primary chromate treatments, comprising 9 proprietary solutions and one solution operated under license were applied to test panels electroplated with the three thicknesses of zinc coating. The treatments represented the various types that are presently on the market. These consisted of two dark-colored treatments used for heavy duty, where protection from corrosion is of primary importance; five bronze or yellow-colored iridescent treatments which are generally used for protection and where appearance is of secondary importance; two bright leached treatments used primarily for appearance; and one electrochemically applied protective treatment.

All proprietary solutions were applied by the respective manufacturers and the solution operated under license was applied by the Industrial Test Laboratory.[5] Prior to application of the supplementary chromate treatments, the electrodeposited zinc test panels were cleaned of surface contaminants by each manufacturer in accordance with his own procedure. After processing, the chromated panels were sealed in polyethylene bags and returned to the Industrial Test Laboratory where they were stored until actual testing was begun. Unchromated zinc panels, similarly cleaned and stored, were included in the tests for comparison purposes. The code letters listed below designate the various chromate treatments applied:

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[3] Federal Specification QQ-Z-325 of 21 January 1952; Zinc Plating (Electrodeposited).

[4] Manufactured by American Instrument Co., Silver Springs, Md.

[5] Philadelphia Naval Shipyard, Philadelphia 12, Pa.

No. 1. *AD*—An iridescent dip treatment in a chromate solution operated under license,

No. 2. *BA*—A yellow-electrolytic treatment,

No. 3. *BD*—A yellow dip treatment,

No. 4. *BE*—A clear dip treatment,

No. 5. *CA*—A bronze dip treatment,

No. 6. *CD*—A clear dip treatment,

No. 7. *DA*—An olive drab dip treatment,

No. 8. *DD*—A yellow dip treatment,

No. 9. *EA*—An iridescent dip treatment, and

No. 10. *EB*—A brown dip treatment.

## METHODS AND RESULTS OF TEST

Test methods employed in this investigation are of two types: long-time exposure tests and accelerated tests. The exposure tests were designed to give information of typical service performance in marine environments. The accelerated tests were investigated as possible methods for quality-control of supplementary chromate films.

### *Exposure Tests*:

Panels were rated for extent of corrosion breakthrough using a weighted numerical rating system from 0 to 30. The panel rating is derived in the following manner:

(*a*) Discoloration and light white corrosion $= \frac{\text{per cent area}}{10} \times 1.$

(*b*) Heavy white corrosion and red rust $= \frac{\text{per cent area}}{10} \times 3.$

The sum of the values obtained for (*a*) and (*b*) denotes the panel rating. It is noted that by this system heavy white corrosion was arbitrarily weighted three times as severe as light corrosion. Red rust was weighted the same as heavy white corrosion since the protec-

TABLE I.—RESULTS OF USS SEVERN EXPOSURE TEST.

| Treatment | Zinc Thickness, mils | Rating[a] | |
|---|---|---|---|
| | | 6 months | 9 months |
| Unchromated zinc | 1.0 | 30 | ..[b] |
| | 0.5 | 30 | ..[b] |
| | 0.2 | 30 | ..[b](E) |
| *AD* | 1.0 | 14 | 18 |
| | 0.5 | 12 | 16 |
| | 0.2 | 15 | 21 (15) |
| *BA* | 1.0 | 26 | 28 |
| | 0.5 | 28 | 30 |
| | 0.2 | 22 | 27 (E) |
| *BD* | 1.0 | 23 | 27 |
| | 0.5 | 24 | 28 |
| | 0.2 | 24 | 27 (5) |
| *BE* | 1.0 | 25 | 28 |
| | 0.5 | 25 | 29 |
| | 0.2 | 26 | 29 (15) |
| *CA* | 1.0 | 19 | 26 |
| | 0.5 | 21 | 26 |
| | 0.2 | 21 | 27 (E) |
| *CD* | 1.0 | 29 | 30 |
| | 0.5 | 28 | 30 |
| | 0.2 | 28 | 30 (10) |
| *DA* | 1.0 | 16 | 21 |
| | 0.5 | 18 | 20 |
| | 0.2 | 10 | 17 |
| *DD* | 1.0 | 26 | 29 |
| | 0.5 | 27 | 30 |
| | 0.2 | 22 | 25 |
| *EA* | 1.0 | 18 | 28 |
| | 0.5 | 24 | 28 |
| | 0.2 | 20 | 26 |
| *EB* | 1.0 | 18 | 24 |
| | 0.5 | 18 | 25 |
| | 0.2 | 19 | 23 |

[a] Ratings reported are the average for 4 replicate panels. Figures in parentheses denote approximate average per cent red rust; (E) denotes red edge rust.

[b] Rating of 30 (100 per cent failure) reached in previous rating period.

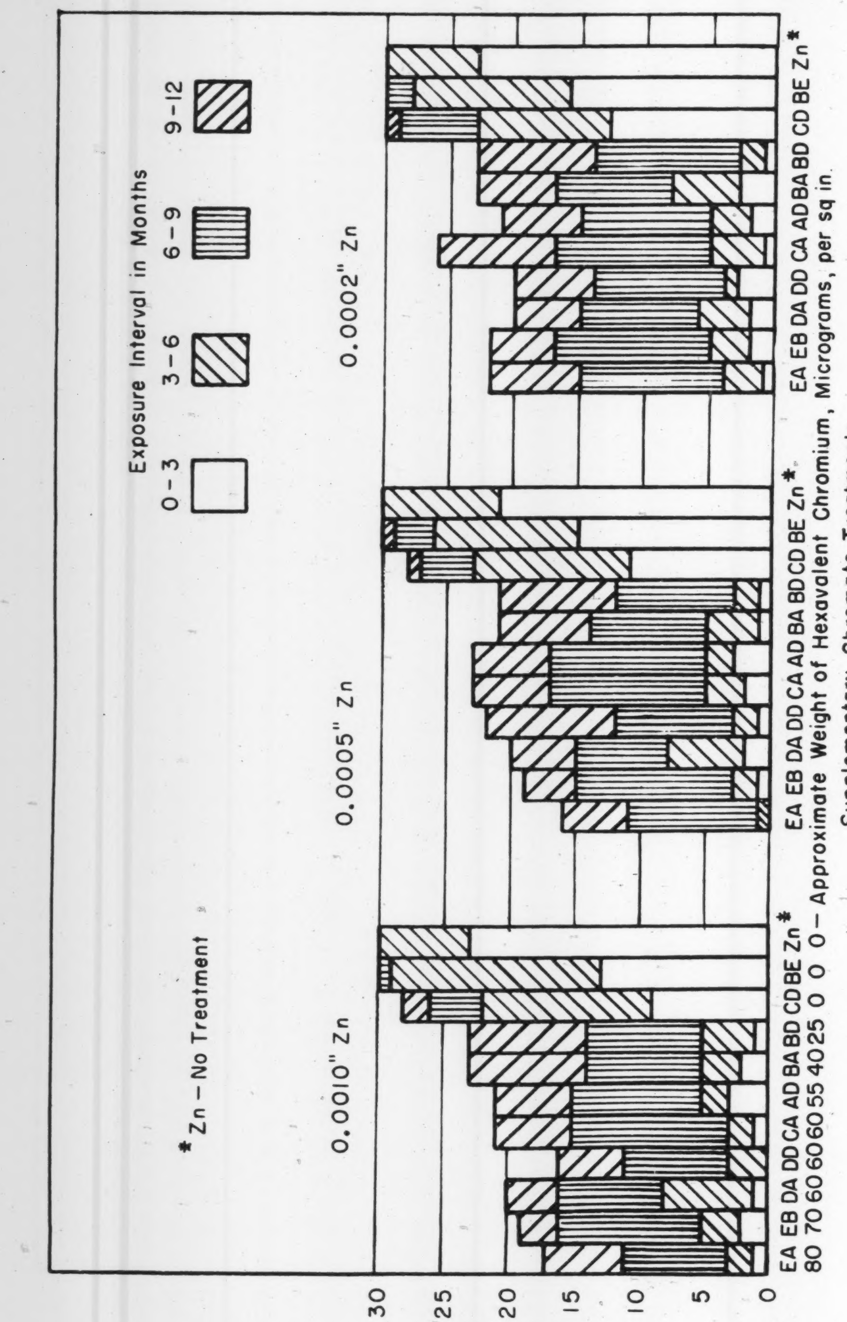

FIG. 1.—Kure Beach Exposure Test (Face Exposure).

tion provided by the treatment and not the zinc is being evaluated.

TABLE II.—RESULTS OF KURE BEACH EXPOSURE TEST.

Figures in parentheses denote approximate average per cent red rust; (E) denotes red edge rust. Ratings reported are the average for 4 replicate panels.

| Treatment | Zinc Thickness, mils | Face Exposure Rating | | | | Underside Exposure Rating | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 3 months | 6 months | 9 months | 12 months | 3 months | 6 months | 9 months | 12 months |
| Unchromated zinc... | 1.0 | 23 | 30 | ..[a] | ..[a] | 19 | 20 | 24 | 24 |
| | 0.5 | 21 | 30 | ..[a] | ..[a] | 15 | 18 | 24 | 25 |
| | 0.2 | 23(5) | 30(20) | ..[a] (45) | ..[a] (65) | 15 | 19 | 23(5) | 25(10) |
| *AD*......... | 1.0 | 3 | 5 | 15 | 21 | 0 | 1 | 3 | 6 |
| | 0.5 | 3 | 5 | 17 | 23 | 0 | 2 | 3 | 5 |
| | 0.2 | 2 | 5 | 15 | 21(E) | 0 | 3 | 3 | 4 |
| *BA*......... | 0.1 | 2 | 5 | 14 | 23 | 0 | 2 | 4 | 6 |
| | 0.5 | 1 | 5 | 14 | 21 | 0 | 3 | 4 | 6 |
| | 0.2 | 3(E) | 8(5) | 17(9) | 23(10) | 0 | 3(E) | 5(4) | 6(5) |
| *BD*......... | 1.0 | 1 | 5 | 14 | 23 | 0 | 3 | 4 | 7 |
| | 0.5 | 1 | 3 | 12 | 21 | 0 | 1 | 2 | 4 |
| | 0.2 | 1 | 3 | 14(E) | 23(8) | 0 | 2 | 3 | 5(E) |
| *BE*......... | 1.0 | 13 | 29 | 30 | ..[a] | 6 | 8 | 12 | 16 |
| | 0.5 | 15 | 26 | 29(5) | 30(15) | 4 | 7 | 12 | 15(E) |
| | 0.2 | 16(7) | 28(20) | 30(65) | ..[a] (85) | 4(E) | 6(5) | 12(8) | 18(10) |
| *CA*......... | 1.0 | 1 | 3 | 15 | 21 | 0 | 1 | 2 | 4 |
| | 0.5 | 2 | 5 | 17 | 23 | 0 | 2 | 4 | 7 |
| | 0.2 | 1 | 5 | 17 | 26(5) | 0 | 3 | 5 | 9(E) |
| *CD*......... | 1.0 | 9 | 22 | 26 | 28 | 4 | 7 | 11 | 14 |
| | 0.5 | 11 | 23 | 27(E) | 28(5) | 4 | 7 | 12 | 12 |
| | 0.2 | 13(10) | 23(15) | 29(50) | 30(75) | 4 | 6(E) | 11(5) | 14(10) |
| *DA*......... | 1.0 | 1 | 8 | 16 | 20 | 0 | 2 | 3 | 4 |
| | 0.5 | 2 | 8 | 15 | 20 | 1 | 2 | 4 | 6 |
| | 0.2 | 2 | 6 | 15 | 20(E) | 1 | 2 | 3 | 5 |
| *DD*......... | 1.0 | 0 | 3 | 11 | 16 | 0 | 1 | 3 | 4 |
| | 0.5 | 1 | 3 | 12 | 22 | 0 | 2 | 3 | 4 |
| | 0.2 | 3 | 4 | 14 | 20 | 1 | 2 | 3 | 5 |
| *EA*......... | 1.0 | 1 | 3 | 11 | 17 | 0 | 0 | 1 | 2 |
| | 0.5 | 0 | 1 | 11 | 16 | 0 | 0 | 1 | 2 |
| | 0.2 | 1 | 4 | 15 | 22(8) | 0 | 1 | 2 | 3(E) |
| *EB*......... | 1.0 | 2 | 5 | 16 | 19 | 0 | 2 | 3 | 6 |
| | 0.5 | 1 | 3 | 15 | 19 | 0 | 2 | 3 | 5 |
| | 0.2 | 2 | 5 | 17 | 22(10) | 0 | 2 | 5 | 9(E) |

[a] Rating of 30 (100 per cent failure) reached in previous rating period.

*Exposure Aboard USS Hyades.*—Four replicate panels (3 by 4 in.) of each chromate treatment and zinc thickness were mounted in a specially designed exposure rack aboard the *USS Hyades*. Four replicates of each thickness of unchromated zinc were included for purposes of comparison. The panels were exposed vertically in randomized positions. The panels had been stored for

2 to 3 months prior to test. The exposure rack was installed in an interior storage room adjacent to the No. 3. refrigeration hold of the ship. This space was believed to be subject to conditions of cyclic condensation and drying. The panels were evaluated for service performance at approximate 6-month intervals. Results of test are summarized as follows: after one year of exposure the panels disclosed no fading of treatment or any indication of deterioration on any specimen including the unchromated zinc panels.

*Exposure Aboard USS Severn.*—A similar set of panels was exposed in an identical manner aboard the *USS Severn.* The panels had been stored for 4 to 5 months. The exposure rack was installed on the weather deck of the ship subject to marine atmospheric conditions. The test panels were evaluated after 6 and 9 months of exposure, following which the test was terminated. Results of exposure evaluations are given in Table I and are summarized as follows:

(*a*) All treatments demonstrate better performance than unchromated zinc. After 9 months of exposure, two treatments, *AD* (iridescent) and *DA* (olive drab) appear to perform the best. Treatment *EB* (brown) appears to perform better than average, and treatment *CD* (clear) appears to be particularly poor. Agreement among replicates was reasonable.

(*b*) Performance of treatments appears to bear no relationship to the thickness of zinc coating to which applied. Red rust was evidenced only on panels of 0.0002 in. zinc coating after 9 months of exposure. Where red rust was observed, the areas were as great or greater on chromated zinc panels than on untreated zinc panels of this thickness.

*Exposure at Kure Beach.*—Four sets of test panels (4 by 6 in.) were exposed on the 80-ft test lot at Kure Beach, N. C. A set consisted of one replicate of each chromate treatment and zinc thickness and one replicate of each thickness of unchromated zinc. Each set was exposed in randomized positions on adjacent frames of the exposure rack. Prior to test, the panels had been stored for 1 to 2 months. Evaluation of the panels for service performance was made at approximately 3-month intervals for a period of one year. The results of test are graphically illustrated in Fig. 1. It is to be noted that the figures below the corresponding treatment code letters represent approximately the average weight of hexavalent chromium for all three thicknesses of zinc coating. The treatments are shown in order of decreasing weight of hexavalent chromium. Results of exposure evaluations for both the face and underside of the panels are given in Table II. The results are summarized as follows:

(*a*) All treatments appear to perform better than unchromated zinc. After one year of exposure, the treatments, except *CD* and *BE* (clear), generally appear to perform alike. The latter appear to show poor performance. On the whole, the degree of failure after 12 months of exposure is not considered as severe as that encountered on the *USS Severn* after 9 months. Also, agreement among replicates was good. Face surfaces demonstrate more deterioration than underside.

(*b*) In general, the performance of the treatments over all thicknesses of zinc coating appears similar. Areas of red rust in most instances were observed to be greater with clear dip treatments, *CD* and *BE*, on 0.0002- and 0.0005-in. zinc coatings than on corresponding thicknesses of unchromated zinc. After 12 months of exposure, the evidence of red rust on colored chromated panels is limited to treatments applied to 0.0002-in. zinc coating.

*Accelerated Tests*:

*Chromium Measurement Tests*:

(*a*) *One and Six Months' Laboratory*

TABLE III.—RESULTS OF CHROMIUM MEASUREMENT TESTS (1 AND 6 MONTHS' LABORATORY AGING).

| Treatment | Zinc Thickness, mils | Chromium, μg per sq in. | | | |
|---|---|---|---|---|---|
| | | Hexavalent Chromium | | Trivalent Chromium | |
| | | 1 month | 6 months | 1 month | 6 months |
| *AD*...... | 1.0 | 54 | 58 | 199 | 228 |
| | 0.5 | 52 | 52 | 237 | 232 |
| | 0.2 | 47 | 52 | 222 | 231 |
| Avg.... | | 51 | 54 | 219 | 230 |
| *BA*...... | 1.0 | 51 | 49 | 119 | 134 |
| | 0.5 | 36 | 33 | 117 | 122 |
| | 0.2 | 41 | 19 | 111 | 136 |
| Avg.... | | 43 | 34 | 116 | 131 |
| *BD*...... | 1.0 | 29 | 22 | 134 | 141 |
| | 0.5 | 27 | 35 | 146 | 148 |
| | 0.2 | 15 | 26 | 125 | 129 |
| Avg.... | | 24 | 28 | 135 | 139 |
| *BE*...... | 1.0 | 0 | 1 | 56 | 54 |
| | 0.5 | 0 | 0 | 42 | 34 |
| | 0.2 | 0 | 0 | 55 | 42 |
| Avg.... | | 0 | 0 | 51 | 43 |
| *CA*...... | 1.0 | 57 | 61 | 209 | 211 |
| | 0.5 | 53 | 58 | 194 | 205 |
| | 0.2 | 63 | 70 | 202 | 219 |
| Aug.... | | 58 | 63 | 201 | 212 |
| *CD*...... | 1.0 | 0 | 1 | 28 | 37 |
| | 0.5 | 0 | 2 | 29 | 31 |
| | 0.2 | 0 | 1 | 23 | 30 |
| Avg.... | | 0 | 1 | 27 | 33 |
| *DA*...... | 1.0 | 59 | 53 | 426 | 439 |
| | 0.5 | 63 | 64 | 415 | 456 |
| | 0.2 | 61 | 48 | 402 | 336 |
| Avg.... | | 61 | 55 | 414 | 410 |
| *DD*...... | 1.0 | 59 | 64 | 203 | 217 |
| | 0.5 | 54 | 51 | 199 | 163 |
| | 0.2 | 64 | 60 | 220 | 217 |
| Avg.... | | 59 | 58 | 207 | 199 |
| *EA*...... | 1.0 | 87 | 78 | 196 | 181 |
| | 0.5 | 74 | 80 | 169 | 198 |
| | 0.2 | 93 | 60 | 219 | 173 |
| Avg.... | | 85 | 73 | 194 | 184 |
| *EB*.... | 1.0 | 90 | 81 | 246 | 267 |
| | 0.5 | 56 | 78 | 242 | 261 |
| | 0.2 | 60 | 53 | 218 | 238 |
| Avg.... | | 69 | 71 | 235 | 255 |

*Aging.*—Two sets of test panels (4 by 6 in.)—a set consisting of one replicate of each chromate treatment and zinc thickness—were removed from the polyethylene bags following receipt of all chromated panels from the manufacturers. The panels of each set were supported vertically in separate grooves notched in a wooden beam and exposed in a laboratory room under dry storage conditions. At the end of one month the bottom ½ in. of one set of panels was sheared and discarded. A 2- by 4- in. test specimen was then sheared from each panel for measurement of hexavalent and trivalent chromium. At the end of 6 months, test specimens were similarly prepared from the other set of panels. The hexavalent chromium content of the films was determined electrophotometrically, employing diphenylcarbizide as the coloring agent. Trivalent chromium was then measured by a variation of the same procedure. The hexavalent chromium was leached from the test specimen by a hot dilute sodium hydroxide solution followed by dissolution of the insoluble trivalent chromium in dilute sulfuric acid. Zinc and iron produced no interference by the method used. The results of these tests are given in Table III and are graphically illustrated in Fig. 2. It is to be noted that the weights of chromium depicted in Fig. 2 are based on averages obtained for three thicknesses of zinc coating including both 1- and 6-month measurement tests. The results are summarized as follows:

1. Treatments aged for one and six months show no systematic difference in hexavalent or trivalent chromium content due to thickness of zinc coating,
2. No loss of chromium is evidenced up to six months aging, and
3. The ratio of trivalent to hexavalent chromium is significantly higher for treatment *DA* (olive drab) than for

other treatments having a similar level of hexavalent chromium.

(*b*) *Depletion Rate of Hexavalent Chromium.*—To study the depletion rate of hexavalent chromium in chromate films exposed to marine atmospheric conditions, four additional replicate sets of panels were exposed on the 80-ft test site at Kure Beach. The panels were mounted on frames adjacent to the panels exposed for evaluation of service performance. The time, procedure, conditions, and test panels, with the exception of the unchromated zinc panels which were eliminated from this test, were the same as for the service performance exposure. At each 3-month inspection interval, one set of panels was removed from the exposure site and measurements made of both hexavalent and trivalent chromium following the procedure described in (*a*). Results of test are given in Table IV. The depletion of hexavalent chromium is graphically illustrated in Fig. 3. It is to be noted that the weights of hexavalent chromium, shown in Fig. 3, are based on the averages obtained for the three thicknesses of zinc coating. The results of test are summarized as follows:

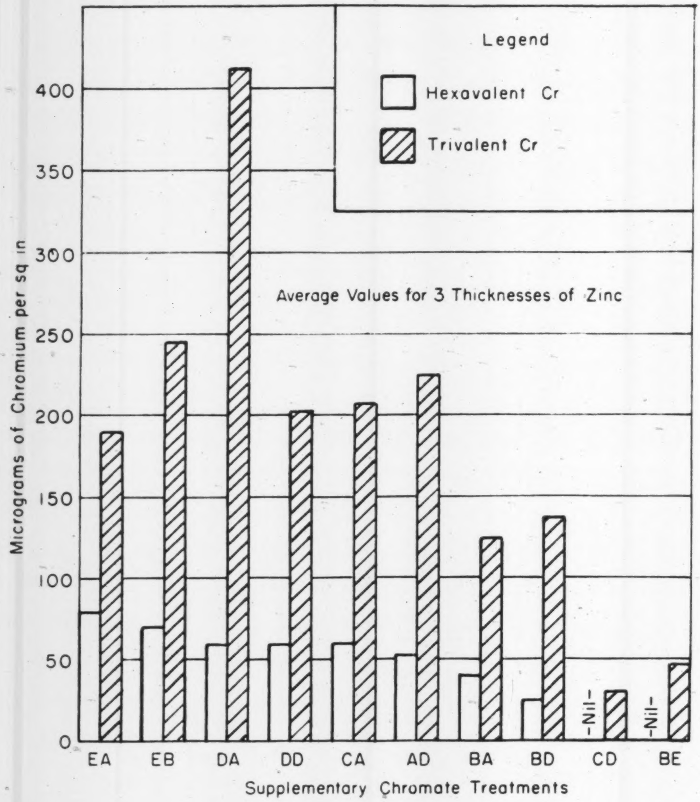

FIG. 2.—Chromium Measurement Tests.

1. Hexavalent chromium depletion increases with time of exposure. However, some treatments appear to show a greater depletion rate than others. Treatment *DA* (olive drab) shows

practically complete loss of hexavalent chromium within three months; treatment *BA* (yellow-electrolytic) appears to show a loss of approximately 85 per cent of its available hexavalent chromium within this same exposure period. The other treatments appear to exhibit a more gradual depletion rate extending over the 12-month exposure period. It appears, however, that in practically all cases only a small proportion of the original hexavalent chromium present in the film is available after 9 months of exposure. No relationship is apparent between the depletion of hexavalent chromium and the thickness of zinc coating to which a treatment was applied.

TABLE IV.—DEPLETION OF HEXAVALENT CHROMIUM—RESULTS OF CHROMIUM MEASUREMENT TESTS AFTER KURE BEACH EXPOSURE.

| Treatment | Zinc Thickness, mils | Chromium, μg per sq in. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Hexavalent Chromium, months exposed | | | | | Trivalent Chromium, months exposed | | | | |
| | | 0[a] | 3 | 6 | 9 | 12 | 0[a] | 3 | 6 | 9 | 12 |
| *AD*............ | 1.0 | 56 | 31 | 19 | 11 | 2 | 214 | 225 | 247 | 169 | 152 |
| | 0.5 | 52 | 26 | 14 | 8 | 2 | 235 | 258 | 252 | 198 | 218 |
| | 0.2 | 50 | 22 | 1 | 4 | 2 | 227 | 257 | 260 | 184 | 169 |
| *BA*............ | 1.0 | 50 | 5 | 3 | 2 | 0 | 127 | 132 | 144 | 145 | 153 |
| | 0.5 | 35 | 6 | 1 | 1 | 0 | 120 | 129 | 133 | 140 | 145 |
| | 0.2 | 30 | 0 | 0 | 1 | 0 | 124 | 129 | 145 | 149 | 150 |
| *BD*............ | 1.0 | 26 | 4 | 6 | 1 | 1 | 138 | 123 | 137 | 138 | 126 |
| | 0.5 | 31 | 9 | 6 | 2 | 1 | 147 | 155 | 148 | 121 | 122 |
| | 0.2 | 21 | 7 | 1 | 2 | 1 | 127 | 149 | 136 | 125 | 127 |
| *BE*............ | 1.0 | 1 | 0 | 0 | 0 | 0 | 55 | 45 | 44 | 43 | 51 |
| | 0.5 | 0 | 0 | 0 | 0 | 0 | 38 | 41 | 47 | 43 | 44 |
| | 0.2 | 0 | 0 | 0 | 0 | 0 | 49 | 46 | 45 | 51 | 49 |
| *CA*............ | 1.0 | 59 | 25 | 7 | 8 | 3 | 210 | 233 | 224 | 224 | 199 |
| | 0.5 | 56 | 17 | 7 | 9 | 4 | 200 | 211 | 196 | 216 | 165 |
| | 0.2 | 67 | 21 | 15 | 10 | 4 | 211 | 221 | 209 | 222 | 186 |
| *CD*............ | 1.0 | 1 | 0 | 0 | 0 | 0 | 33 | 29 | 27 | 33 | 29 |
| | 0.5 | 1 | 0 | 0 | 0 | 0 | 30 | 28 | 29 | 31 | 33 |
| | 0.2 | 1 | 0 | 0 | 0 | 0 | 27 | 28 | 27 | 29 | 24 |
| *DA*............ | 1.0 | 56 | 1 | 0 | 0 | 0 | 433 | 331 | 233 | 141 | 100 |
| | 0.5 | 64 | 1 | 0 | 0 | 0 | 436 | 350 | 203 | 166 | 177 |
| | 0.2 | 55 | 0 | 0 | 0 | 0 | 369 | 295 | 187 | 122 | 114 |
| *DD*............ | 1.0 | 62 | 29 | 17 | 9 | 10 | 210 | 207 | 193 | 195 | 169 |
| | 0.5 | 53 | 22 | 18 | 3 | 1 | 181 | 223 | 227 | 201 | 179 |
| | 0.2 | 62 | 24 | 15 | 8 | 5 | 219 | 227 | 234 | 166 | 186 |
| *EA*............ | 1.0 | 83 | 67 | 33 | 2 | 2 | 189 | 229 | 205 | 193 | 192 |
| | 0.5 | 77 | 36 | 42 | 2 | 1 | 184 | 221 | 215 | 221 | 155 |
| | 0.2 | 77 | 28 | 14 | 2 | 2 | 196 | 216 | 202 | 196 | 131 |
| *EB*............ | 1.0 | 86 | 31 | 22 | 7 | 1 | 257 | 291 | 282 | 252 | 218 |
| | 0.5 | 67 | 21 | 30 | 19 | 1 | 252 | 295 | 331 | 279 | 187 |
| | 0.2 | 57 | 19 | 17 | 12 | 1 | 228 | 281 | 272 | 202 | 172 |

[a] Average of chromium measurements for 1 and 6 months' aging tests.

2. For all treatments, trivalent chromium measurements indicate, with the exception of treatment *DA* (olive drab), very little loss of trivalent chromium over the entire 12-month exposure period. Treatment *DA* appears to exhibit an appreciable loss of trivalent chromium over each 3-month testing interval.

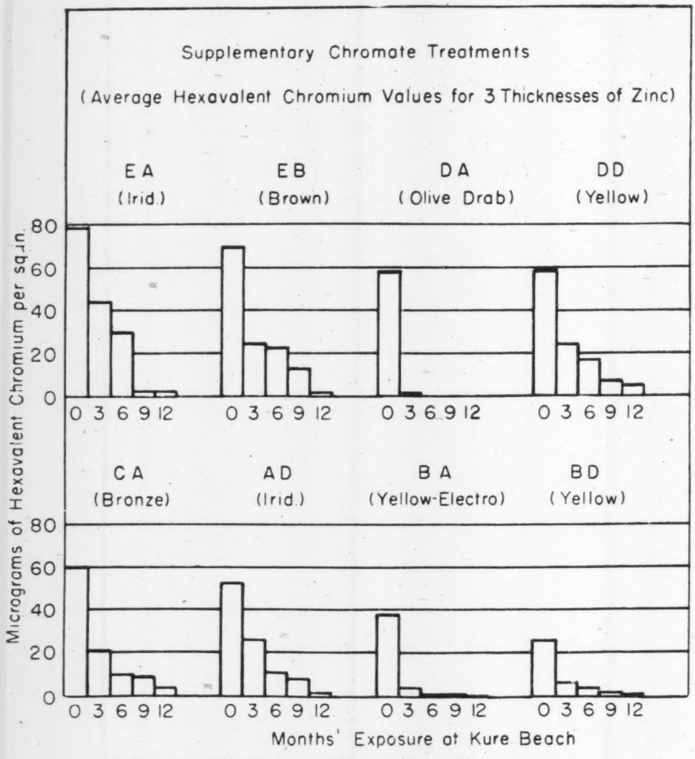

FIG. 3.—Depletion of Hexavalent Chromium.

*Spot Tests*:

Five spot tests generally employed by the industry were investigated. Two of these showed initial promise in this work. The basis of the spot testing procedure is the dissolving action of the testing solution on the chromate film. This is accompanied by a color change denoting the end point. The time for a change in color to occur is taken as a criterion of film quality and is noted as "reaction time." The following solutions were used in test:

*Solution A*.—A proprietary solution consisting of two separate chemical solutions mixed in equal volume just prior to use. The color changes from light amber to blue-green.

*Solution B*.—A 5 per cent solution of lead acetate. The color changes from colorless to blue-black. In this test, the color begins to change at the periphery of the drop, and the end point is denoted by a complete change in color.

(*a*) *One- and-Six Months' Laboratory Aging Tests*.—Spot tests were performed on the unused portions of panels remaining from the chromium measurement tests after one and six months of

TABLE V.—RESULTS OF SPOT TESTS (1 AND 6 MONTHS OF LABORATORY AGING).

Results are based on average of 4 or more determinations over both sides of panel.

Average reaction time of solution A on unchromated zinc was $\frac{1}{4}$ min.

Average reaction time of solution B on unchromated zinc was $\frac{3}{4}$ min.

| Treatment | Zinc Thickness, mils | Reaction Time, min | | | |
|---|---|---|---|---|---|
| | | Solution A | | Solution B | |
| | | 1 month | 6 months | 1 month | 6 months |
| *AD*...... | 1.0 | 14 | 13 | 21 | 14.5 |
| | 0.5 | 17 | 15 | 28 | 27.5 |
| | 0.2 | 18 | 12 | 30 | 20.5 |
| *BA*...... | 1.0 | 1 | 0.5 | 21 | 5.5 |
| | 0.5 | 1 | 0.5 | 9 | 6.5 |
| | 0.2 | 1 | 0.5 | 10 | 6 |
| *BD*...... | 1.0 | 8.5 | 6 | 34 | 7.5 |
| | 0.5 | 13 | 4.5 | 15.5 | 8.5 |
| | 0.2 | 6 | 6 | 12 | 10 |
| *BE*...... | 1.0 | 2.5 | 3 | 2.5 | 3 |
| | 0.5 | 1.5 | 1.5 | 2 | 2.5 |
| | 0.2 | 1.5 | 1.5 | 2 | 2.5 |
| *CA*...... | 1.0 | 10 | 3.5 | 20 | 7.5 |
| | 0.5 | 4.5 | 4 | 9 | 10 |
| | 0.2 | 8.5 | 7 | 15.5 | 13 |
| *CD*...... | 1.0 | 1 | 1 | 2 | 2 |
| | 0.5 | 1 | 1 | 2 | 1.5 |
| | 0.2 | 1 | 1 | 1.5 | 1.5 |
| *DA*...... | 1.0 | 16 | 4.5 | 29 | 2 |
| | 0.5 | 22.5 | 5 | 33.5 | 3.5 |
| | 0.2 | 12.5 | 15 | 14.5 | 14.5 |
| *DD*...... | 1.0 | 12 | 6.5 | 31.5 | 9.5 |
| | 0.5 | 15 | 3 | 57 | 5 |
| | 0.2 | 9 | 7 | 41 | 19.5 |
| *EA*...... | 1.0 | 16 | 12 | 23.5 | 12 |
| | 0.5 | 21.5 | 12.5 | 24 | 11.5 |
| | 0.2 | 14.5 | 9 | 17.5 | 10.5 |
| *EB*...... | 1.0 | 6 | 3.5 | 10.5 | 3 |
| | 0.5 | 10 | 4 | 13 | 3.5 |
| | 0.2 | 16 | 13.5 | 5 | 10.5 |

aging in open racks in the Laboratory. Results of the test are given in Table V and are summarized as follows:

(*1*) A study of the data indicates insufficient agreement between the results obtained with Solutions *A* and *B* for alignment of treatments in order of merit. The performance of treatment *BA* (yellow-electrolytic) appears to be particularly poor with Solution *A*. The clear dip treatments, *BE* and *CD*, show poor performance with both spot testing solutions.

(*2*) Tests on colored chromate films over the three thicknesses of zinc coating generally show large and erratic differences in reaction time.

(*3*) Panels aged for 6 months generally show a significant drop in reaction time compared to panels aged for one month.

(*4*) Agreement among replicate determinations by both spot testing procedures was poor for treatments demonstrating a higher average reaction time level.

*Salt Spray (Fog) Test:*

This fog test was conducted in accordance with ASTM Method B117[6] employing a 20 per cent sodium chloride solution. A set of test panels (4 by 6 in.), comprising one replicate of each treatment and zinc thickness, was exposed in a single salt spray box. Unchromated zinc panels were included for purposes of comparison. Prior to test, the panels had been stored for approximately 7 months. The panels were exposed in randomized positions in wooden racks. At 24-hr intervals the panels were inspected and the racks rotated. The treatments were rated for number of days to appearance of initial white corrosion products and presence of 50 per cent white corrosion products. The data shown in Table VI represent the average results of 4 replicate determinations performed at different intervals. These data are also graphically illustrated in

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[6] Tentative Method of Salt Spray (Fog) Testing (B 117 - 54 T), 1955 Book of ASTM Standards, Part 2, p. 871; Part 4, p. 668.

Fig. 4. The results are summarized as follows:

(*a*) Presence of initial white corrosion products can occur from within 1 to 10 days and 50 per cent white corrosion products from within 3 to 17 days depending upon the treatment. All treatments improve the salt spray resistance of zinc-plated steel surfaces.

(*b*) Comparison of the results of the initial and 50 per cent white corrosion tests indicates that, except for *EA* (iridescent), all treatments can be aligned in a similar order of merit. Treatment *EA* appears to exhibit the best salt spray resistance in the 50 per cent white corrosion test, whereas in the initial white corrosion test its performance is only average. The clear dip treatments, *CD* and *BE*, show the poorest performance.

TABLE VI.—RESULTS OF SALT SPRAY FOG TESTS.

Results reported are the average for 4 replicate panels.

| Treatment | Days to Initial White Corrosion[a] Zinc Thickness, mils | | | Days to 50 per cent White Corrosion Zinc Thickness, mils | | |
|---|---|---|---|---|---|---|
| | 1.0 | 0.5 | 0.2 | 1.0 | 0.5 | 0.2 |
| Unchromated zinc | 0 | 0 | 0 | 1.0 | 1.0 | 1.0 |
| *AD* | 5.7 | 5.7 | 5.5 | 12.7 | 9.7 | 9.7 |
| *BA* | 4.5 | 3.0 | 4.0 | 9.7 | 6.0 | 7.0 |
| *BD* | 2.2 | 4.2 | 4.7 | 10.5 | 9.2 | 8.2 |
| *BE* | 1.5 | 1.5 | 1.7 | 6.5 | 5.5 | 5.0 |
| *CA* | 6.0 | 5.7 | 5.5 | 11.0 | 11.0 | 9.0 |
| *CD* | 1.2 | 1.2 | 1.2 | 5.0 | 4.7 | 4.7 |
| *DA* | 7.0 | 6.5 | 4.2 | 13.0 | 12.0 | 8.7 |
| *DD* | 4.7 | 5.2 | 4.2 | 10.0 | 10.5 | 7.5 |
| *EA* | 5.7 | 5.2 | 4.7 | 16.0 | 14.5 | 10.7 |
| *EB* | 7.7 | 7.5 | 5.2 | 13.7 | 13.5 | 9.7 |

[a] Edge failures excluded.

(*c*) Salt spray resistance of treatments to the appearance of initial white corrosion products appears similar over all thicknesses of zinc coating.

## DISCUSSION

Results of various exposure tests demonstrate, in general, that supplementary chromate treatments afford protection to electroplated zinc coatings in marine environments. They also reveal that the clear films provide less protection against corrosion than the colored chromate films. Based on theoretical considerations, this would indicate that both hexavalent and trivalent chromium play a role in the usefulness of the film and that hexavalent chromium is the controlling factor. The Kure Beach exposure test (Fig. 1) discloses that films having a minimum content of 25 $\mu$g of hexavalent chromium per square inch provide relatively good protection for approximately 6 months. After 6 months of exposure, the films deteriorate more rapidly but still retain corrosion inhibiting properties.

A study of the depletion of hexavalent chromium (Fig. 3) was made simultaneously with the service performance test at Kure Beach. The results indicate that for most treatments hexavalent chromium ceases to be a major factor in retarding corrosion after approximately 6 months. This assumption is supported by the significant increase in deterioration of the panels after this length of

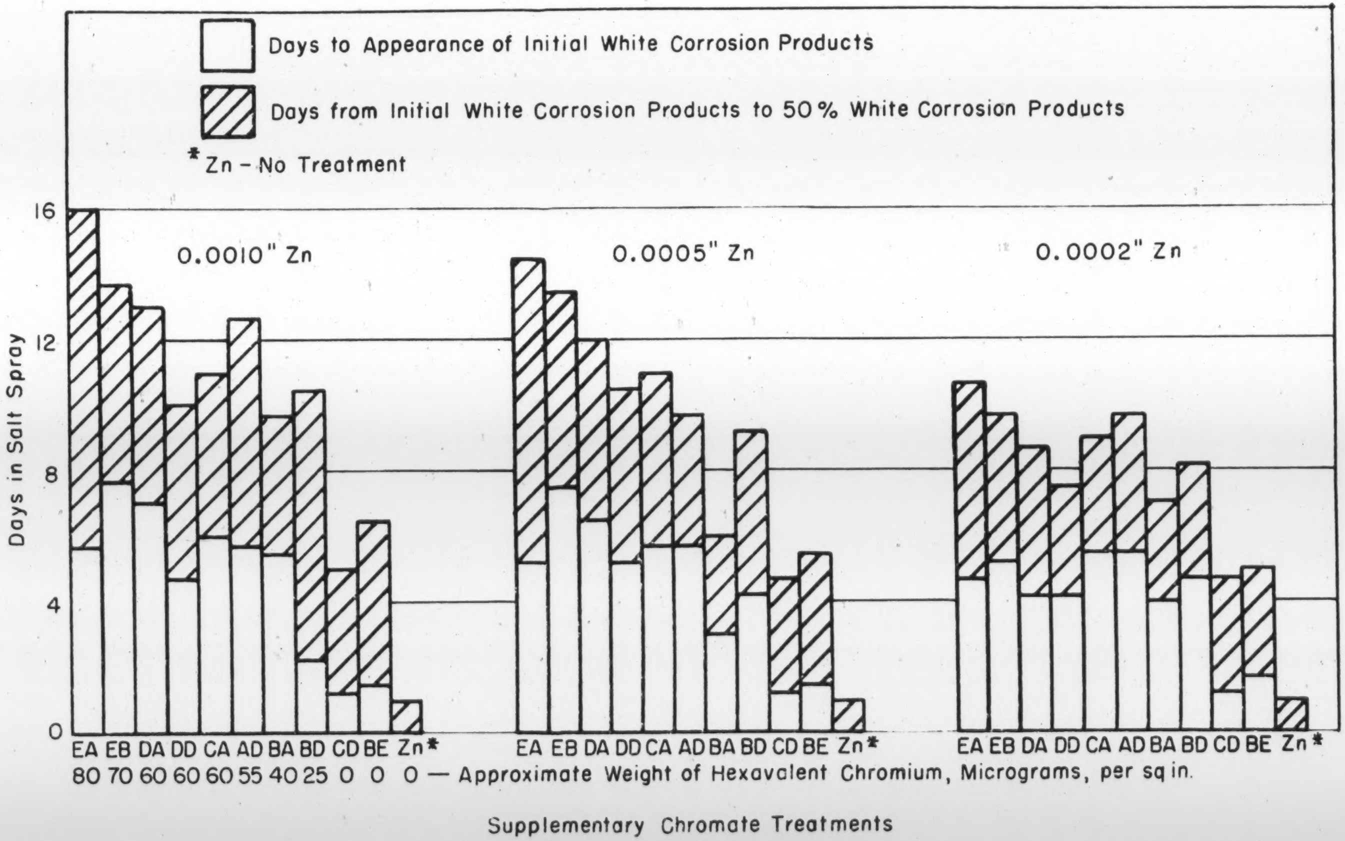

FIG. 4.—Salt Spray Fog Tests.

exposure. The relatively large increase in deterioration of treatment *DA* (olive drab) after 3 months of exposure can also be attributed to the practically total depletion of hexavalent chromium within 3 months.

It was observed that deterioration of the chromated panels, following the depletion of available hexavalent chromium, did not progress as rapidly as the panels to which no treatment was applied. The explanation of this phenomenon may lie in the corrosion inhibiting properties of the trivalent chromium ($Cr_2O_3$) part of the film. This insoluble trivalent chromium appears to contribute substantially to the protection. Thus, the protection afforded by the clear dip treatments can be assumed to be entirely provided by the relatively small trivalent chromium content of the films.

Much attention was centered on the performance of treatment *DA* (olive drab) relative to exposure testing and loss of chromium on exposure. This treatment exhibited unusual characteristics compared to other treatments employed in test. Determination of initial chromium present in the film disclosed a rather high hexavalent chromium content and a ratio of trivalent to hexavalent chromium of approximately 7 to 1 (Fig. 2). The average ratio for the other treatments tested was about 3 or 4 to 1. The available hexavalent chromium was depleted in 3 months and the high trivalent chromium demonstrated an appreciable loss over each 3-month period. Exposure tests indicated that notwithstanding the premature depletion of its hexavalent chromium and substantial losses of trivalent chromium, the film deteriorated at a fairly moderate rate and continued to exhibit corrosion inhibiting properties for the duration of the tests. In some instances, treatments showing less deterioration than treatment *DA* after 6 months of exposure exhibited a greater degree of deterioration than *DA* after 9 and 12 months.

Correlation between the exposure tests performed at Kure Beach and on the *USS Severn* is difficult to assess. Treatments *AD* (iridescent) and *DA* (olive drab) demonstrated the best performance in the *Severn* test and average performance at Kure Beach. Treatment *EB* (brown) performed better than average and clear dip treatments, *CD* and *BE*, poorly in both exposures. The inferior performance of treatments *DD* (yellow) and *EA* (iridescent) in the *Severn* test in relation to their performance at Kure Beach lacks an adequate explanation. All results indicate that the Severn test manifests a more severe exposure condition than the Kure Beach test.

Investigation of accelerated tests was undertaken with the objective of acquiring standard methods for the quality-control of supplementary chromate films on zinc. Various spot test data demonstrate that this method of testing is unfeasible for this purpose. Experience with spot tests indicates they are better employed for establishing the presence or absence of a chromate treatment than for differentiating between films of varying qualities. Several factors, notably age of chromate films and variations of reaction time between replicate determinations, tend to make spot tests unreliable for quality-control.

Based on exposure testing at Kure Beach, it appears that determination of the weight of hexavalent chromium in the film offers a feasible method for quality-control. Age of films and thickness of zinc coating to which the treatments are applied present no difficulties in the use of the method. Analysis of fairly large size replicate specimens generally yielded reproducible results. The Kure Beach test (Fig. 1) demonstrates that films containing an initial hexavalent chromium weight of 25 to 80 μg per sq in. perform fairly much alike. Therefore, a minimum requirement for hexavalent chromium of 25 μg per sq in. of film would insure the rejection of clear dip treatments and the acceptance of chromated films having necessary corrosion inhibiting properties. Moreover, the hexavalent chromium requirement would also usually be accompanied by more than three times as much trivalent chromium (Fig. 2) which also contributes substantially to the protection.

The salt spray fog test also appears to offer a feasible method for quality-control. It is indicated that the controlling factor in resistance to salt spray is the level of hexavalent chromium present in the film (Fig. 4). Whereas the Kure Beach exposure test reveals only slight differences in performance of colored treatments relative to the level of hexavalent chromium content, the salt spray tests tend to demonstrate more significant differences. However, both exposure and salt spray tests show measurable differences in performance between colored and clear treatments. Results of the salt spray tests indicate that time to appearance of initial white corrosion products correlates reasonably with time to 50 per cent white corrosion products. For quality-control purposes, therefore, it would be more expedient to denote the end point of salt spray testing as the appearance of white corrosion products, exclusive of edge corrosion. A minimum of 4 days in 20 per cent salt spray before the end point is reached can be taken as a criterion for acceptance of chromated zinc.

The Kure Beach and *USS Severn* exposure tests indicate that the effectiveness of chromate treatments in affording protection to zinc in marine environments is approximately alike, irrespective of the thickness of zinc coating to which they are applied. However, after 12 months of exposure at Kure Beach and 9 months of exposure on the *Severn* areas of red rust on colored chromated panels were observed only on those electroplated with 0.0002 in. zinc. Based on this observation it is apparent that after the chromate films become ineffective for providing protection to the zinc, the thickness of the remaining zinc coating is the controlling factor in affording protection to the basis metal.

In the Kure Beach exposure test, the areas of red rust observed on 0.0002- and 0.0005-in. zinc panels to which clear dip treatments had been applied, in most instances, were greater than on corresponding thicknesses of untreated zinc panels. After 9 months of exposure on the *Severn*, 0.0002-in. zinc panels having clear dip films and, in certain instances, colored chromate films were observed to show greater areas of red rust compared to untreated zinc panels of similar thickness. These observations substantiate the commonly known fact that some surface attack of the zinc coating occurs during application of the chromate treatment, thereby reducing the thickness of the original coating and thus lessening the protection provided by the zinc. It is therefore indicated that to insure adequate over-all protection, supplementary chromate treatments should be applied to a zinc coating thickness in the order of 0.0005-in or greater.

Following the above periods of exposure at Kure Beach and on the *USS Severn* all treatments over all thicknesses of zinc showed greater than 50 per cent deterioration, indicating that no further necessary information could be acquired by continued exposure.

## CONCLUSIONS

1. Application of supplementary chromate treatments over electrodeposited zinc coatings on steel inhibit corrosion of the zinc in marine environments.

2. The soluble hexavalent chromium content of the film is the primary factor in providing protection against corrosion, assisted by the insoluble trivalent chromium portion which contributes substantially to the protection.

3. Chromate supplementary films containing a minimum weight of 25 $\mu$g per sq in. of hexavalent chromium afford the best protection in marine environments.

4. Supplementary chromate treatments are effective over all thicknesses of zinc coating. However, due to the surface attack of zinc incurred during application of a chromate treatment, the protection provided by the zinc to the basis metal is reduced. A thickness of 0.0002-in. zinc coating for application of supplementary chromate treatments is, therefore, considered inadequate.

5. Determination of hexavalent chromium content and the salt spray (fog) test can be employed as accelerated tests for quality-control of chromate supplementary films. Spot tests can establish the presence or absence of a film but are unreliable for quality-control purposes.

# REPORT OF COMMITTEE B-9
## ON
# METAL POWDERS AND METAL POWDER PRODUCTS*

Committee B-9 on Metal Powders and Metal Powder Products held one meeting during the year: in Philadelphia, Pa., on February 5, 1957.

The committee consists of 73 members, of whom 40 are classified as producers, 24 as consumers, and 9 as general interest members.

Mr. John B. Haertlein was appointed chairman of Subcommittee II on Metal Powders, and Section A on Base Metal Powders, succeeding J. J. Cordiano who resigned these chairmanships.

Subcommittee III-D on Friction Materials has been reactivated with the appointment of Howard B. Huntress as Chairman.

### TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued without revision in view of pending changes:

*Tentative Specifications for:*

Sintered Metal Powder Structural Parts from Bronze (**B 255 - 55 T**), and

Sintered Structural Parts from Brass (**B 282 - 53 T**).

*Tentative Method of:*

Sieve Analysis of Granular Metal Powders by Air Classification (**B 293 - 54 T**).

*Tentative Recommended Practice for:*

Hardness Testing of Cemented Carbides (**B 294 - 54 T**).

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Nomenclature and Technical Data* (F. N. Rhines, chairman) prepared definitions for such terms as "K-factor," "radial crushing strength," "infiltration," and revisions of the definition for the term "impregnation."

A review is under way of the status of the term "sintering," used in defining a product made by sintering metal powders. The program is continuing of gathering technical data relative to specifications, such as B 222 - 52 for Sintered Metal Powder Structural Parts.

*Subcommittee II on Metal Powders* (J. B. Haertlein, Chairman):

Section A on Base Metal Powders (J. B. Haertlein, chairman) is working on a new method for the compressibility of metal powders, and methods for testing the dimensional change of iron sintered in a standard bar. Committee E-3 on Chemical Analysis of Metals has been requested to develop methods of chemical analysis for metal powders. The subcommittee recommended the continuance as tentative of the Method of Subsieve Analysis of Granular Metal Powders by Air Classification (B 293-54 T) to allow additional time for the general use of the method.

Section B on Refractory Metal Powders (W. H. Bleecker, chairman).—Task groups are at work on chemical analysis, turbidimetric analysis, and microscopic analysis as well as on a tentative method of test for apparent density of refractory metals and compounds by means of the Scott volumeter. There has recently been completed a proposed Tentative Method for Average Particle Size of Refractory Metals and Compounds by the Fisher Subsieve-Sizer which has been referred to letter ballot of the subcommittee.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

*Subcommittee III on Metal Powder Products* (R. P. Koehring, chairman):

Section A on Bearings (M. H. Meighan, chairman) is studying the correlation of machinability, microstructure, and K-factor of bronze bearings falling within the scope of B 202 – 55 T, Tentative Specification for Metal Powder Sintered Bearings (Oil Impregnated) as well as standards for sintered flange bearings and a revision of type II class B of Specification B 202 – 55 T. When the proposed tentative for the determination of density and porosity is approved as a separate test method, this will lead to revision of all the specifications where such a test is now a part of the specifications. A task force was appointed to work with Committee D-2 on Petroleum Products and Lubricants with respect to recommended lubricants for sintered bearings.

Section B on Structural Parts (P. J. Shipe, chairman) has recommended to Committee B-9 the following changes in specifications: Deletion of the note on hardness testing from Specifications B 222 – 52 T, B 225 – 54 T and B 282 – 53 T that states hardness values are considered unreliable; and removal of the ten-day limitation for the rejection of parts where it appears in any specification; and revision of the Appendix in Specification B 282 – 53 T for Sintered Metal Powder Structural Parts from Brass.

This committee has prepared a tentative test method for determining the density and porosity of sintered metal parts which has been submitted to Committee B-9 for approval.

Section C on Cemented Carbides (E. W. Engle, chairman) has recommended the continuance as tentative of B 294 – 54 T, Recommended Practice for Hardness Testing of Cemented Carbides, until the results of round robin hardness tests are obtained which prove good agreement in values. The section has prepared a draft recommended practice for determination of transverse rupture strength of cemented carbides which has been submitted to the subcommittee for approval.

Section D on Friction Materials (H. B. Huntress, chairman) has reviewed a number of facets relative to preparing specifications and test methods for friction materials, and work has been started on test methods for evaluating the physical and mechanical properties of friction materials. An effort will be made to develop test methods for friction testing and evaluation.

Section E on Machinable Heavy Metals (A. S. Doty, chairman) is continuing work on a specification for machinable high-density tungsten-nickel-copper alloys.

This report has been submitted to a letter ballot of the committee, which consists of 74 members; 60 members returned their ballots, of whom 51 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. L. BONANNO,
*Chairman.*

C. G. JOHNSON,
*Secretary.*

# REPORT OF ADVISORY COMMITTEE
# ON
# CORROSION*

The Advisory Committee on Corrosion held two meetings during the year: on November 1, 1956, at ASTM Headquarters, and on February 6, 1957, during Committee Week in Philadelphia, Pa.

## PERSONNEL

Committee D-20 on Plastics was asked to be represented on the Advisory Committee and has appointed F. W. Reinhart and H. A. Perry as representatives. New committee representatives are as follows: for Committee A-3, D. E. Krause and H. W. Stuart; Committee A-5, Marc Darrin and H. F. Hormann; Committee B-5, G. F. Geiger; and Committee C-19, George Gerard. The Advisory Committee learned with regret of the passing of W. A. Kennedy, Chairman of Committee A-7 on Malleable Iron Castings, on April 9.

H. A. Pray was recommended for reappointment as a member-at-large for a three-year term from June 1957.

The officers elected for the ensuing term of two years are as follows:

Chairman, K. G. Compton.

First Vice-Chairman, C. P. Larrabee.

Second Vice-Chairman, W. H. Finkeldey.

S. F. Etris was appointed as ASTM Staff Secretary to the Advisory Committee.

## STATUS OF ATMOSPHERIC EXPOSURE PROGRAMS

*Committee A-5 on Corrosion of Iron and Steel:*

The 20-yr inspection and collection of wire and fencing test specimens was made during the summer of 1956. The data from the nine exposure test sites will be combined and published in a report of 20 years of atmospheric exposure of wire specimens.

*Committee A-7 on Malleable Iron Castings:*

A series of 1620 plates have been prepared. These include 600 standard malleable, 600 pearlitic malleable, 240 nodular iron, and 180 steel. Half of the cast plates will be exposed with completely machined surfaces, half with as-cast surfaces. The pearlitic malleable plates will be of both low and high combined carbon types. One group of nodular plates is as-cast, the other heat-treated. The steel plates are structural, copper bearing, and alloy. These specimens will be placed at Point Reyes, Calif., East Chicago, Ind., New York Area (Newark, N. J.), Kure Beach, N. C., and State College, Pa.

*Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:*

Approximately 5000 specimens of sheets, bar, wire, and coil springs plus various weldments of all grades of currently produced flat rolled products will be placed at four exposure test sites during the spring of 1958. The objective of this series of tests is to obtain comprehensive data on the performance of the several grades of stainless steel in typical atmospheric environments. The test site locations will be State College, Pa., New York Area (Newark, N. J.), Kure Beach, N. C., and Freeport, Tex.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

*Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:*

A new atmospheric exposure test program is being set up to obtain information on the galvanic behavior of magnesium alloys coupled to other materials. It is hoped that this new program will permit the committee to devise a galvanic test sample which will be better than the disc-type couple now being used. Three sets of panels will be exposed concurrently at four test sites.

*Committee B-7 on Light Metals and Alloys, Cast and Wrought:*

The five-year exposure specimens of aluminum and magnesium alloys at five test sites have been collected and are now being tested. Data will be published in the 1958 Annual Report of Committee B-7.

*Committee B-8 on Electrodeposited Metallic Coatings:*

Another group of massive cadmium specimens may be added to the previous estimate of 216 cadmium-plated panels and 42 massive panels to be set out at each of three test sites: Rock Island, Ill., New York Area (Bell Telephone Laboratories, New York), and Kure Beach, N. C. These will be placed for exposure this spring.

*Committee C-19 on Structural Sandwich Constructions:*

The sandwich panels of the first committee program, after two years' exposure at Kure Beach, N. C., and State College, Pa., have been removed for testing. This spring, the second program began with the exposure of 72 additional panels at these same two test sites.

## TEST SITE LEASES

The Advisory Committee has been extending the leases of sites where Committee A-5 has had wire fencing exposure tests in progress for the past 20 years. The test site leases that are being extended for ten more years are: Ames, Iowa; College Station, Tex.; Davis and Santa Cruz, Calif.; Ithaca, N. Y.; Lafayette, Ind.; and Manhattan, Kans.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
*Chairman.*

S. F. ETRIS,
*Secretary.*

# REPORT OF THE JOINT ASTM-ASME COMMITTEE
ON
# EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS[*]

Meetings of the Joint ASTM-ASME Committee on the Effect of Temperature on the Properties of Metals, its panels, and subcommittees were held during the year in connection with the Annual Meetings of the ASTM in Atlantic City, N. J., June 18 to 22, 1956, and of the ASME in New York, N. Y., November 26 to 30, 1956. All the panels met at both annual meetings.

### *Technical Sessions and Papers:*

The following papers were presented at sessions sponsored by the joint committee:

*Annual Meeting of the ASTM:*

"Materials for Helical Compression Springs for Use at Constant Deflection from 600 to 1400 F," by R. G. Matters and R. E. Lochen.[1]

"Influence of Hot-Working Conditions on the High-Temperature Strength Properties of a Heat-Resistant Alloy," by J. W. Freeman and J. F. Ewing.[2]

*Annual Meeting of the ASME:*

"Basis for the Design and Retirement of Petroleum Heater Tubes," by J. J. Heller.

"Properties of Cast Iron at Elevated Temperatures," by J. R. Kattus.

"The Effect of Elastic Follow-up and Restressing on the Service Life of Chromium-Molybdenum-Vanadium Bolts at High Temperature," by C. Dale Dickinson and Robert Matters.

"Influence of Heat Treatment on the 1000 and 1050 F Properties of Steam Pipe Made in Japan and Germany From 1.25 Cr–0.50 Mo and 2.25 Cr–1.0 Mo Steels," by I. A. Rohrig and J. W. Freeman.

"Chromium-Molybdenum and Chromium-Molybdenum-Vanadium Steels for Power Plant and Refinery Service up to 1100 F," by G. V. Smith.

### *Membership:*

The committee records with sorrow the death of Prof. A. E. White, a member of long standing who was also a member of the Edison Electric Institute's Prime Movers Committee. The committee also regrets the resignation of Irving Roberts who was the chairman of the Low Temperature Panel.

### *Finances:*

The financial status of the Joint Committee is as follows:

| | | |
|---|---|---|
| Cash on Hand, June 30, 1956 | $36 846.38 | |
| Receipts, July 1, 1956 to June 30, 1957 | 2 389.33 | |
| Total | 39 235.71 | |
| Disbursements, July 1, 1956 to June 30, 1957 | 8 597.62 | |
| Cash on Hand, June 30, 1957 | 30 638.09 | |
| Obligated and Unspent | 21 500.00 | |
| | | 9 138.09 |
| Appropriations to Date: | | |
| Data and Publications Panel | 39 200.00 | |
| General Research Panel | 7 000.00 | |
| Steam Power Panel | 1 038.26 | |
| Test Methods Panel | 1 500.00 | |
| Estimated Unallocated Balance June 30, 1957 | | 9 138.09 |

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[*] Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 672 (1956).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 756 (1956).

*Appropriations:*

| | |
|---|---|
| *Data and Publications Panel:* | |
| Completion of Project DP-8, Survey of Medium-Carbon, High-Chromium, and Plain Carbon Steels | $1000.00 |
| DP-11 Current and Continuing Data Survey, and DP-13 Punch Card Tabulation of Data | 2000.00 |
| *General Research Panel:* | |
| Survey on Creep Damage | 2000.00 |
| Survey on Effect of Cyclic Loading and Cyclic Heating on the Creep and Rupture Life of Metals | 2000.00 |
| Survey on Effect of Notches on the Creep-Rupture Properties of Metals | 3000.00 |
| *Steam Power Panel:* | |
| Completion of Project SP-2, Elevated Temperature Properties of Cast Iron | 5000.00 |
| Project SP-5, Investigation of Brittle Conditions Austenitic Stainless Steels | 5000.00 |
| *Test Methods Panel:* | |
| Evaluation of Type 316 Stainless Steel | 1500.00 |

## ACTIVITIES OF PANELS

*Aviation Panel (Leo Schapiro, chairman):*

The problem of accumulating the usable data on elevated temperature properties of titanium alloys was studied, and the Defense Department was asked to have its Titanium Metallurgical Laboratory contractor perform this task. Their report is expected to be issued in May.

Engineering properties of all metal honeycomb sandwich pnaels needed by the aircraft industry and what test methods will best measure these properties were studied. The task group report to the next Panel meeting may complete this study.

The problem of elevated temperature creep testing with compression loading, bearing loading and shear loading was studied. A task group report of several test methods extant is being further studied to determine whether a "best" test method can be recommended.

Structural cermets have been discussed and a task group is assembling compositions and properties of interest to the aviation industry.

The problem of properties with aerodynamic heating has also been discussed, but this wide open area has not yet been resolved to a manageable project.

*Data and Publications Panel (George V. Smith, chairman):*

Project DP-5 has been completed with publication of *ASTM STP 187*, Relaxation Properties of Steels and Super Strength Alloys at Elevated Temperatures. Data collected on Wrought Medium-Carbon Alloy Steels under DP-8 have been published as *ASTM STP No. 199*.

Data collected on weldments under DP-6 and on High-Chromium Steels under DP-8 are being organized for publication. Data are being accumulated under DP-9 on aluminum and magnesium alloys. Surveys are being made of Physical Properties at Low and High Temperatures under DP-10 and of Test Facilities under DP-12.

Project DP-11 concerns development of a scheme for gathering current data as they are obtained, and progress is being made toward this end. Project DP-13 concerns punch-card tabulation of data and its feasibility is being examined.

*Gas Turbine Panel (C. T. Evans, chairman):*

Mr. Herbenar was appointed chairman of Project GTP-10, Long-Time Creep and Stress Rupture Properties, to replace Mr. Olcott. Recent stress rupture failures of Nimonic 80 buckets after about 16,000 hr and 19,000 hr service in pipe line compressor gas turbines were discussed. These failures point up the importance of this project.

Project GTP-2 on Corrosion-Erosion has been reorganized under the chairmanship of Bruce Buckland, other project appointees being Gayley, Margulis, and Kihlgren. A U. S. Navy Bureau of Ships representative will also be requested. Mr. Buckland asked the chairman to point out to the main committee the fact that there are numerous other groups working on the problem of corrosion of high temperature components from residual fuel oil products, and he is in need of guidance as to how the GTP-2 activity can best be fitted into the picture. Other ASME, ASTM, and NACE committees are currently active, and Mr. Buckland would appreciate information on the nature and extent of these studies.

The Data and Publications Panel is considering a survey of the properties of cermets. The Gas Turbine Panel believes that these materials may have a place in future turbine designs and herewith endorses the idea of such a survey.

Mr. Dyrkacz presented a written progress report on Thermal Shock Studies at Allegheny Ludlum showing that N155 and Hastelloy X have the best resistance of the wrought materials tested. The panel recommended that this work be presented as a paper at the annual meeting of the ASME this year.

The most active projects of this panel are as follows: GTP-1, Fatigue; GTP-2, Corrosion-Erosion; GTP-5, Thermal Shock; and GTP-10, Long-Time Creep and Rupture Testing.

*General Research Panel* (*J. D. Lubahn, chairman*):

The panel had a half-day meeting on June 21, 1956 and an all-day meeting on November 8, 1956. The principal item of business at both meetings was the determination of desirable research programs to be carried out under sponsorship by the panel. At the November 8 meeting, nine general subjects, pertaining, for the most part, to elevated temperature behavior, were listed and discussed. Three of these, namely, creep damage, effect of cyclic loading and cyclic heating on creep and rupture life, and effect of notches on creep-rupture properties, were singled out for special consideration. In order to be in a better position to state what research is needed in these fields, the panel authorized literature surveys to be made in all three areas. The survey on the first subject is being conducted at the University of Michigan under the direction of J. W. Freeman. The other two surveys are being done under contract with Syracuse University, the second under the direction of S. S. Manson of NACA with the assistance of G. Sachs of Syracuse University, and the third under the direction of Dr. Sachs. The reports of these surveys will be available for consideration at the next meeting of the panel which will be on June 18, 1957 in Atlantic City.

The panel is also making an effort to extend its membership to include more people with background in Mechanics and Mechanical Engineering so as to have a better balance between the mechanical and metallurgical aspects of materials problems.

At the November 8 meeting, the panel also reviewed and restated its objectives and functions. It was agreed that the panel should recognize and clearly state questions of broad interest to the Joint Committee which may be answered by suitable investigations, determine the most desirable projects to obtain the information needed and initiate appropriate investigations, and support general symposia of interest to the Joint Committee. In order better to carry out these functions, particularly those which pertain to the Joint Committee as a whole, the panel has assigned one of its members to serve as liaison representative for the General Research Panel on each of the other panels of the Joint Committee.

*Low Temperature Panel (Allan Tarr, chairman):*

LTP-2. Circulation of copies of data compilations among selected panel members and surveys to include data on weld metal and weld joints, low-temperature data on fatigue properties of titanium and coefficient of expansion data at low temperature were continued.

LTP-3. Initial work includes analysis of pertinent data, especially determination of strain rates encountered with Charpy V notch and keyhole-notch impact tests at U. S. Army Corps of Engineers, Fort Belvoir, Va., and a study to correlate V-notch and keyhole-notch impact test data on materials used for pressure vessels and piping service.

LTP-4. Initial work includes a study of pertinent data put out by Subcommittee 26, on Low-Temperature Properties of Cast Iron, of ASTM Committee A-3.

The chairman of the panel promoted a three-day conference at Fort Belvoir on welding engineering which should furnish data pertinent to current projects. Consideration was given to the preparation of a proposal for the panel to sponsor an ASTM Symposium on applications of stress at high rates of strain with special reference to the influence of low temperature and weld joint discontinuity in densities of structural elements.

*Petroleum and Chemical Panel (C. L. Clark, chairman):*

The report prepared by W. B. Hoyt on "The Properties of Carbon Steel Weldments at Elevated Temperatures" was reviewed in detail at the November 26 meeting and, with one minor revision, was approved for passing on to the Main Committee for possible presentation at the ASME meeting the coming year. The revision consists of including in the paper a sketch showing the position of the plates during welding and the location of the specimens used for the tests.

Mr. Hoyt reported that similar work on alloy steel weldments is now well along, and a corresponding report should be available either at the June ASTM meeting or the December ASME meeting.

*Steam Power Panel (P. M. Brister, chairman):*

SP-1. Past activity on the subject of steam and air oxidation tests of low-alloy ferritic steels to 1100 F was discussed, but the panel refused to authorize any expenditure of funds to conduct accurate oxidation tests and suggested a cooperative program of field testing in operating boilers. It was thought that the Steam Generation Committee studies of this subject might adequately cover the interests of the panel.

SP-2. Progress was reported on the elevated temperature properties of cast iron being carried out at Southern Research Inst. Rupture tests at 5000 hr have been started on the two most promising cast irons, and thermal shock tests have been conducted on seven of the irons.

SP-4 (Elevated Temperature Properties of SA-212B Plate). It was reported that the creep and rupture tests were progressing satisfactorily, and most of the creep testing is completed.

SP-5 (Use of Austenitic Steels for Steam Line Service). The present work appears to be very promising as a means of classifying good or bad material, but, to date, little or no work has been done on studies to determine the fundamental reasons which make a material good or bad. It was requested that a planned study program aimed at determining the fundamentals involved be inaugurated. There appeared to be a need for further tests on materials from service to demonstrate the ability of the tests conducted to separate the crack-sensitive from the noncrack-sensitive materials.

It was recommended that the panel be used as a coordinating agent for reviewing new materials to determine if sufficient information is available on them for commercial use. However, there was no action taken on this recommendation.

*Test Methods Panel* (*J. J. Kanter, chairman*):

The panel has under way a program for providing standard test materials for calibrating creep and rupture test equipment. The use of calibration test specimens had been suggested as a possible means of obviating the necessity for defining the precision of temperature measurement and control in testing of this sort. At the last meeting, agreement was reached on the type of steel to be used for the calibration specimens and on the general program for establishing properties. A heat of type 316 steel (about 10,000 lb) has been melted and is currently being rolled to $\frac{15}{16}$-in. round cornered square bars as per a procedure agreed upon by the task force. After heat treatment, the material will be tested to determine uniformity of properties. The National Bureau of Standards has been approached relative to this phase of the work for which an appropriation of $1500 was approved.

The three Recommended Practices under jurisdiction of the Joint Committee, namely Short Time Elevated Temperature Tension Tests of Metallic Materials (E 21), conducting Long Time High-Temperature Tension Tests of Metallic Materials (E 22), and conducting Time-for-Rupture Tension Tests of Metallic Materials (E 85) are in need of revision and a Task Force under J. W. Freeman is currently engaged in this work. This group will also decide whether notched stress rupture testing should be included in Recommended Practice E 85. Recommendations as to revisions of the three practices are expected to be available for discussion at the June meeting of the panel.

A new campaign for funds is to be made, the goal being $100,000. This is to be undertaken by the Finance Committee under the chairmanship of Mr. Norman Mochel.

Respectfully submitted on behalf of the committee,

V. T. MALCOLM,
*Chairman.*
J. J. KANTER,
*Vice-Chairman.*

HOWARD C. CROSS,
*Secretary.*

# REPORT OF COMMITTEE C-1
## ON
# CEMENT*

Committee C-1 on Cement held two meetings during the year: in Atlantic City, N. J., on June 21, 1956, and in Los Angeles, Calif., on September 18, 1956.

The committee records with sorrow the death of H. F. Kichline, a member since 1933, who was the representative of the North American Cement Corp.

At the 1956 Annual Meeting of the Society two members of the committee were honored by the Society: H. F. Gonnerman, Honorary Member of Committee C-1, received the ASTM Award of Merit, and F. H. Jackson was recognized as a 40-year member of the Society.

### Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1956 Annual Meeting, the committee presented to the Society, through the Administrative Committee on Standards, the recommendation that the Tentative Specifications for Masonry Cement (C 91 – 55 T) be revised.

This recommendation was accepted by the Standards Committee on December 17, 1956, and the revised tentative specifications appear in the 1956 Supplement to the Book of ASTM Standards, Part 3.

### Revision of Tentatives

The committee recommends revisions as indicated of the following tentative specifications and methods and their continuation as tentative:

**Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205 – 56 T):**[1]

*Section 2(a).*—Change to read as follows:

2(*a*). *Type IS.*—For the purpose of these specifications, portland blast-furnace slag cement is the product obtained by intimately intergrinding a mixture of portland cement clinker and granulated blast-furnace slag. The portland cement clinker as that from which portland cement as described in the Standard Specifications for Portland Cement (ASTM Designation: C 150) can be made. The amount of granulated blast-furnace slag used is such that the slag constituent makes up between 25 and 65 per cent by weight of the portland blast-furnace slag cement.

*New Section.*—Add a new Section 2(*b*) as follows, relettering the present Section 2(*b*) as 2(*c*):

2(*b*). *Type IS-A.*—Air-entraining portland blast-furnace slag cement is the product obtained by intimately intergrinding a mixture of portland cement clinker, and granulated blast-furnace slag. Sufficient air-entraining addition as specified in Section 3(*c*) shall be interground with the clinker and blast-furnace slag to meet the air content requirement of Table II. The portland cement clinker is that from which portland cement as described in the Standard Specifications for Portland Cement (ASTM Designation: C 150) can be made. The amount of granulated blast-furnace slag used is such that the slag constituent makes up between 25 and 65 per cent by weight of the portland blast-furnace slag cement.

*Section 4(b).*—Change the opening sentence to read, "The composition of the granulated blast-furnace slag constituent shall conform to the requirements of the following formula:"

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1956 Supplement to Book of ASTM Standards, Part 3.

*Section 6(a).*—Change to read as follows:

(*a*) At the request of the purchaser, the manufacturer shall state in writing the source, amount, and composition of the granulated blast-furnace slag used in the finished cement, the composition of the portland cement clinker constituent, and the composition of the portland blast-furnace slag cement of the type purchased.

*Table II—Physical Requirements.*—Opposite the words "Air content of mortar, prepared and tested in accordance with Method C 185, per cent by volume" insert in the column for Type IS the words "less than 12.", and in the column for Type IS-A change "16 ± 4" to read "19 ± 3."

### Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cement (C 230 – 55 T):[2]

*Fig. 1.*—Change the legend "2-Shafts Medium carbon machinery steel" to read "1-Shaft Medium carbon machinery steel," and delete the phrase above this legend which reads "Approx $1\frac{3}{4}''$ adjusted to give drop of $\frac{1}{8}'' \pm 0.005''$." This action is recommended because there are no current ASTM specifications which utilize the $\frac{1}{8}$-in. drop.

### Tentative Method of Test for Bleeding of Cement Pastes and Mortars (C 243 – 55 T):[2]

*New Footnote.*—Add a new footnote 5 as follows and renumber the subsequent footnotes accordingly; add a reference to the new footnote 5 at the end of Section 2(d):

[5] Attention is called to the desirability of using certain accessory equipment in order to increase speed and reproducibility such as a centering ring for the collecting ring, a templet for setting the collecting ring to the proper depth, and a plastic, two-piece cover to minimize drying. These accessories are furnished by some manufacturers of the bleeding equipment.

*Section 2(d).*—Change the ninth sentence to read:

"The rim of the funnel shall be ground on a plane parallel to the axis of the funnel and buret but the resulting edge shall be rounded." This change will bring the description into conformance with the actual apparatus.

*Section 2(e).*—Change to read as follows:

(*e*) *Mechanical Mixer.*—A mechanical mixer shall be used which conforms to the requirements of Tentative Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (ASTM Designation: C 305) except that:

(*1*) A first or low speed ranging from 135 to 150 rpm is permitted.[6]

(*2*) The mixing bowl may be tin-plated or stainless steel and shall have a nominal capacity of not less than 3 qt nor more than 5 qt.

(*3*) A paddle of material other than stainless steel may be used.

The above changes are necessary to continue the acceptability of the Hobart Model C-10 Mixer as well as the Hobart Model N-50 Mixer for use in bleeding tests.

*Fig. 1.*—Change the value "$2\frac{11}{16}$" now designated as the small diameter of the collecting ring, to read "$2\frac{5}{8}$," and change the circular inset in the figure to show the edge of the funnel slightly rounded instead of knife-edged. These changes are necessary to bring Fig. 1 into conformance with the dimensions of the bleeding apparatus as currently manufactured, and to conform to the recommended revision of Section 2(*d*) in regard to the edge of the funnel.

### Tentative Specifications for Portland-Pozzolan Cement (C 340 – 55 T):[2]

*Section 1.*—Change to read as follows:

1. These specifications cover two types of hydraulic cement, as follows:

*Type IP.*—Portland-Pozzolan Cement.

*Type IP-A.*—Air-entraining Portland-Pozzolan Cement.

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[2] 1955 Book of ASTM Standards, Part 3.

*Section 3.*—Change Paragraph (*a*) to read as follows, retaining the present Note to this section:

> (a) *Type IP.*—For the purpose of these specifications, portland-pozzolan cement is the product obtained by intimately intergrinding a mixture of portland cement clinker and pozzolan, or an intimate and uniform blend of portland cement and fine pozzolan. The portland cement clinker is that from which portland cement as described in the Standard Specifications for Portland Cement (ASTM Designation: C 150) can be made. The pozzolan constituent shall conform to the physical requirements prescribed in Section 7. The amount of pozzolan used is such that the pozzolan constituent makes up between 15 and 50 per cent by weight of the portland-pozzolan cement.

Add a new Paragraph (*b*) to read as follows, relettering the present Paragraph (*b*) as Paragraph (*c*):

> (*b*) *Type IP-A.*—Air-entraining portland-pozzolan cement is the product obtained by intimately intergrinding a mixture of portland cement clinker and pozzolan, or an intimate and uniform blend of portland cement and fine pozzolan. Sufficient air-entraining addition as specified in Section 4(*c*) shall be interground with the clinker and pozzolan constituent to meet the air content requirement of Table II. The portland cement clinker is that from which portland cement as described in the Standard Specifications for Portland Cement (ASTM Designation: C 150) can be made. The amount of pozzolan used is such that the pozzolan constituent makes up between 15 and 50 per cent, by weight, of the portland-pozzolan cement.

*Section 4.*—Redesignate Paragraph (*c*) of Footnote 4 as new Paragraph (*c*) for Section 4.

*Table II.—Physical Requirements.*—Opposite the words "Air content of mortar prepared and tested in accordance with Method C 185, per cent by volume" in the column for Type IP-A change "18 ± 3" to read "19 ± 3."

### Tentative Specifications for Slag Cement (C 358 – 55 T):[2]

*Table II.—Physical Requirements.*—Opposite the words "Air content of mortar prepared and tested in accordance with Method C 185, per cent by volume" in the column for Type SA change "18 ± 3" to read "19 ± 3."

## TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions of the Standard Methods of Chemical Analysis of Portland Cement (**C 114 – 53**)[2] as appended hereto.[3]

## ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Masonry Cement (**C 91 – 56 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentative specifications and methods of test be continued as tentative without revision. All are subjects of current study within the committee.

*Tentative Specifications for:*

Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (**C 226 – 54 T**).

*Tentative Method of Test for:*

Potential Alkali Reactivity of Cement-Aggregate Combinations (**C 227 – 52 T**),
Time of Setting of Hydraulic Cement by Gillmore Needles (**C 266 – 51 T**),
Flexural Strength of Hydraulic Cement Mortars (**C 348 – 54 T**), and
Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (**C 349 – 54 T**).

## EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

### Tentative Specifications for Masonry Cement (C 91 – 56 T):[1]

*Section 28(b).*—At the end of the

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[3] These tentative revisions have been incorporated in the Tentative Methods of Chemical Analysis of Portland Cement (C 114 – 57 T) which appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

second sentence insert the words "at medium speed."

### Standard Methods of Chemical Analysis of Portland Cement ( 114-53):[2]

*Section 20 (a).*—Add the following note at the end of this paragraph:

NOTE.—These methods are not considered to be suitable for the determination of the loss of volatile matter on ignition of portland blast-furnace slag cement and of slag cement. A method suitable for such cements is described in Section 19 of the Tentative Methods for Chemical Analysis of Portland Cement (ASTM Designation: C 114 - 57 T), 1957 Supplement to Book of ASTM Standards, Part 3.

### Standard Method of Test for Tensile Strength of Hydraulic Cement Mortars (C 190 - 49):[2]

*Section 2(f).*—Change to read: "The trowel shall be of a convenient size and have a steel blade 4 to 6 in. long." This change gives a more helpful description of the trowel.

*Section 2(g).*—Add the following note:

NOTE.—For instructions relating to the manner and frequency of calibration of the testing machine see Sections 49 and 58 of the Manual of Cement Testing, Appendix II, ASTM Standards on Cement (With Related Information).

*Section 8.*—Add the following note:

NOTE.—For instructions relating to the cleanliness and frequency of changing of the storage water see Section 15 of the Manual of Cement Testing, Appendix II, ASTM Standards on Cement (With Related Information).

### Tentative Method of Test for Bleeding of Cement Pastes and Mortars (C 243 - 55 T):[2]

*Section 1.*—Change to read: "This method of test for determining the bleeding rate and bleeding capacity of cement paste and mortar by a direct and continuous procedure is intended for use solely in research."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

### SPECIAL REPORT BY SUBCOMMITTEE

A special report of the Subcommittee on the Cement Reference Laboratory entitled "History of the Cement Reference Laboratory, 1929–1955" is appended hereto.[5] This interesting report on the conception, development, and accomplishments of one of the committee's best known projects was assembled by John R. Dwyer, supervisor of the project over the period covered by the report, who was acting on behalf of the subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 91 voting members; 72 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
*Chairman.*

W. S. WEAVER,
*Secretary.*

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[5] See p. 232.

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### EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Revision of Tentative Method of Test for:*

Calcium Sulfate in Hydrated Portland Cement Mortar (C 265 - 56 T)
Flexural Strength of Hydraulic Cement Mortars (C 348 - T)
Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (C 349 - 54 T)

These recommendations were accepted by the Standards Committee on December 13, 1957, and the revised tentative methods appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

## APPENDIX I

# HISTORY OF THE CEMENT REFERENCE LABORATORY 1929–1955

## REPORT OF SUBCOMMITTEE ON CEMENT REFERENCE LABORATORY[1]

Occasional published reports have described the Cement Reference Laboratory (CRL)—its purpose, establishment, procedure, and accomplishments. However, some of those reports are now quite old, and more recent changes in specifications and tests have made some of the earlier information of little current importance. Accordingly, there is now offered a somewhat complete, but not too detailed, narrative of the CRL from the formative period to about the end of its 10th inspection tour (the early part of 1955). References indicated in the text will enable the reader to pursue some items in more detail if he so desires.

### EVENTS LEADING TO THE PROPOSAL OF THE PROJECT

*Reasons.*—In the early part of the century, a number of organizations collaborated in the endeavors to improve and standardize the specifications and test methods for portland cement. Among those organizations were three that were formed at just about the same time and which have collaborated ever since: The Portland Cement Assn., the National Bureau of Standards, and ASTM. The work of ASTM in the cement field became chiefly centered in its Committee C-1 on Cement. That committee was organized in 1902, and in its ranks were staff members of both the Portland Cement Assn. and the National Bureau of Standards.

For a long time Committee C-1 and other groups cooperated in the endeavors to attain closer agreement in the results of tests of cement by different operators and by different laboratories. Cement standards were made more exacting. Comparative tests were conducted by groups of laboratories. However, progress was not satisfactory, and the problem became even more pressing as changes occurred in the test methods and in the cements themselves.

In 1926, when reporting on an extensive cooperative study of strength tests, the chairman of the Subcommittee on Strength (a subcommittee of Committee C-1) stated: "It is conclusively shown that where a number of laboratories make the same type of either tension or compression test pieces from the same cement and sand, according to methods as clearly described as the committee has been able to outline them, so great a variance of results is obtained among the laboratories that it would seem very unlikely that any definite recommendations could be made as to the preferred test piece or method, until some means had been evolved to train more uniformly skilled operators."

*Plans.*—Later investigations only emphasized the problem, and Committee C-1 appointed a special committee to bring about the establishment of a laboratory that would, through field work and otherwise, demonstrate proper performance of standard methods, calibrate testing apparatus, and promote uniformity in the testing of cement. The special committee consisted of Frederick W. Kelley (Chairman), Cloyd M. Chapman, and Richard L. Humphrey. Frederick Kelley, then President of the North American Cement Corp., had long been connected with cement manufacture and had been active in the technical work of both the Portland Cement Assn. and Committee C-1. In the latter group, he had been the ardent proponent of such a special laboratory. Cloyd Chapman was active in cement and concrete fields, and later (1932) was to serve as President of ASTM. Richard Humphrey had served as Secretary of Committee C-1 from its founding in 1902 to 1916, and he had been Secretary of ASTM in that Society's formative years (1898-1900).

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[1] Material assembled by J. R. Dwyer, Consultant, Cement Reference Laboratory.

## ESTABLISHMENT OF THE LABORATORY

*Assistance.*—The Special Committee visited the Secretary of Commerce, the Director of the National Bureau of Standards, and the Director of the Budget. Success rewarded the committee's efforts to make suitable arrangements. One-half of the funds was provided through a special contribution from the Portland Cement Assn. to ASTM. Following the committee's appeal to the Bureau of the Budget, the other half of the necessary money was provided in the form of a Government appropriation of funds for the National Bureau of Standards (**1**).[2]

In the establishment of this project, the Special Committee received valuable help and advice from numerous collaborators, among whom were: George K. Burgess, Director of the National Bureau of Standards, who had been President of ASTM in 1922–23; G. W. Thompson, then President of ASTM; William M. Kinney and George E. Warren, respectively General Manager and Assistant General Manager of the Portland Cement Assn.; C. L. Warwick, Executive Secretary of ASTM; P. H. Bates, who was then Chairman of Committee C-1 on Cement and also Chief of Division 9 of the National Bureau of Standards.

*Sponsorship and Support.*—Under the sponsorship of Committee C-1, the new project was quickly established at the National Bureau of Standards. Under the name Cement Reference Laboratory, this unique cooperative project of the Government and ASTM began work in April, 1929, with J. R. Dwyer in charge, operating within Division 9 of the NBS. The original financial supporters were later joined by the U. S. Bureau of Public Roads and the U. S. Corps of Engineers. Through the years the funds from the industry were received through ASTM. From the very beginning of the work, ASTM has generously assisted in handling the various non-Governmental funds, arranging details of insurance, furnishing helpful advice, planning for related meetings, and absorbing numerous expenses connected with the work. Through the assistance of the various supporters and collaborators, the CRL has been enabled to work continuously since 1929 (**1, 3, 4, 5**).

*Technical Assistant to Committee C-1.*—In 1932, Committee C-1 established a new position—that of Technical Assistant to Committee C-1. The intent of the new project was to facilitate, by travel and correspondence, the interchange of information on cement specifications, promote uniformity in such specifications, and aid in the work of

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[2] The boldface numbers in parentheses refer to the list of references at the end of this report, see p. 247.

Committee C-1. Since 1932, the Head of the CRL has acted as the Technical Assistant. Through such arrangement, most of the secretarial work of the committee has centered in the CRL, thus bringing about a very close relationship between the activity of the CRL and its sponsor, Committee C-1 on Cement.

*Personnel and Equipment.*—The CRL staff, always small, has varied in size as circumstances and funds dictated. The staff has been made up in part by Government employees and in part by men paid from the funds made available through ASTM. The latter men are classed as Research Associates, and they work under the regulations governing Research Associates at the National Bureau of Standards. The Bureau has furnished most of the clerical services, as well as laboratory facilities and office space, and much of the equipment necessary for the work.

*Supervision.*—The CRL has always operated within Section 6 (Concreting Materials) of Division 9 of the National Bureau of Standards. All correspondence and operations have been conducted in accordance with applicable Bureau regulations. The immediate direction of the work has been the responsibility of the Head of the CRL. The laboratory has regularly reported to the Chief of Section 9.6 at the Bureau, and to the Subcommittee on Cement Reference Laboratory (a subcommittee of Committee C-1). The CRL Subcommittee has ever been helpful in providing counsel and assistance.

## SCOPE OF ACTIVITIES OF THE CRL

*Outline.*—The Laboratory's work may be outlined briefly under the following headings:

Inspection of apparatus.
Instructions in test methods.
Inspections of cement testing laboratories.
Distribution of comparative test samples.
Special tests of cements for Committee C-1.
Cooperation with subcommittees of Committee C-1.
Secretarial work for Committee C-1.

*General Conditions.*—The laboratory has no police authority, and does not attempt to impose its services. Cement laboratories are inspected only upon their request. No apparatus is "rejected." No certification or rating is given to a laboratory. The CRL has tested certain special lots of cements for Committee C-1 when such materials were involved in specification considerations by the committee, but no tests are ever made on general acceptance samples of cement, or on cement samples related to disputes.

*Progress Reports.*—Some of the Annual Reports of Committee C-1, as published in the ASTM *Proceedings*, have related many details of the nature and extent of the laboratory's accomplishments (**1, 4, 7, 11, 13**). Occasional articles in the ASTM BULLETIN discussed the work (**2, 3, 9**), as did also a detailed report presented by P. H. Bates and J. R. Dwyer at one of the technical meetings of the Portland Cement Assn. in 1930 (**5**). However, it seems appropriate to summarize in considerable detail the laboratory's work during the period of ten inspection tours, approximately 1929 to 1955, in order to convey reminders of the purpose of the laboratory, outline the scope and amount of its work, and present data that may be helpful in the future.

## INSPECTION WORK

*Inspection of Apparatus.*—The inspection of apparatus is largely limited to equipment required by current ASTM cement tests, but additional tests are sometimes made on apparatus that may be of particular interest to Committee

C-1 or the CRL. The apparatus is examined for general condition and for compliance with the detail requirements of applicable ASTM standards. Practically all of the inspections of apparatus are made in the course of the inspection visits to the many cement laboratories throughout the country, although a small amount of apparatus has been received for test at the Bureau. Reports of test of apparatus mention whether such apparatus meets, or fails to meet specification requirements, but no statements of "rejection" are made. Where feasible, each piece of satisfactory apparatus has a permanent CRL identifying number imposed thereon at the time of its first inspection.

Some of the more decided variations from standard that were encountered in the earliest examinations of apparatus may have been occasioned by the absence of applicable tolerances to indicate how closely a manufacturer should adhere to nominal specification requirements, or how well a laboratory should maintain its equipment. One of the earliest tasks in planning the work of the inspectors was the selection of appropriate tolerances by Committee C-1 (**1, 2**). In the meetings and correspondence leading to the selection of such tolerances, the committee had the benefit of advice from both makers and users of cement testing equipment. Also, the CRL collected pertinent information from various sources, particularly from shops and laboratories in the National Bureau of Standards. Special facilities at the Bureau enabled the CRL to study the effect of some of the variables, and to become acquainted with the procedures required in the inspection of equipment.

*Demonstrations of Methods of Test.*—Instructions in the standard methods of testing cement have been occasionally given to operators who visited the Bureau to receive such help where there is the opportunity to see considerable equipment and procedures related to their work. However, most of the instruction on methods has been accomplished in the course of the inspection tours among the many cement laboratories.

*Inspection of Cement Laboratories.*—The CRL has devoted the major part of its endeavors to the inspection of cement laboratories, and to the related correspondence and confirmatory reports. It is important to remember that the CRL does not seek to impose its services, but visits a laboratory only when requested to do so. However, voluntary participation in this inspection service has been widespread and quite continuous. Inspection itineraries have been arranged so that interested laboratories could be visited at intervals of about three years. These periodic inspections have been furnished without charge to the inspected laboratories, but nearly all of the laboratories are those in which the various supporters of the CRL are interested, directly or indirectly.

The inspection of laboratories includes the tests of the apparatus required by the standard tests of cement, the inspection of methods, demonstrations of tests by the inspector when warranted, examination of facilities for storage of test specimens, and collection of miscellaneous pertinent information.

Along with the increase in the amount of verification work on compression testing machines that were used in the tests of standard mortar cubes, there arose numerous requests for the verification of the larger machines employed in the strength tests of concrete. Conditions did not permit extending the verification service to laboratories other than those engaged in testing cement, but it was considered that the strength tests of concrete were very closely involved in the testing of cement in cement laboratories. Accordingly, the work among the cement laboratories was enlarged to include the verification of their compression testing machines up to a load of 200,000 lb. Also, observations were made concerning the curing conditions, molds, plates, and capping procedure involved in the tests of concrete cylinders in those laboratories.

*Reports of Inspection of Laboratories.*—Upon the completion of his work in a laboratory, the inspector confers with an appropriate member of the laboratory staff, making available to him the data on the inspector's work sheets, and discussing all important items that may have been observed in the course of the visit.

No rating or certification is given to a laboratory, but a detailed confirmatory report is sent to the official who requested the inspection. The inspector's findings and the confirmatory report are confidential. However, copies of the confirmatory reports have sometimes been made available to interested offices upon their request, provided the issuance of such copies has been authorized by the inspected laboratory. Some Governmental agencies have availed themselves of such procedure in the case of laboratories in which they were interested. In any case, decision as to the acceptability or selection of a cement laboratory for certain work must be made by the supervising official involved—never by the CRL. All distribution of reports is made by the CRL.

The confirmatory report has often been the basis upon which a laboratory has secured new equipment. Also, the data and queries resultant from the laboratory inspections sometimes include material of great interest to some of the C-1 subcommittees, to whom appropriate information is transmitted.

*Inspector's Travel, Equipment, and Test Procedure.*—At the commencement of the work, the inspectors traveled by train, transporting their then simple equipment as baggage. A few years later they traveled in their personally-owned cars, carrying a slightly increased lot of apparatus. Still later, the addition of proving rings made considerable increase in the bulk and weight of the equipment, and the inspectors have since traveled in light panel trucks of the Bureau. Apparatus has been added as required by specification developments, and the present-day field outfit includes the following items: weights, balance, various gages, thermometers, psychrometers; calibration lever and weights for checking the tensile machines; proving rings of various capacities up to 200,000 lb, together with the necessary bearing blocks; equipment for testing pressure gages on autoclaves; microammeter test sets; miscellaneous related tools and minor equipment.

The changes in cement testing since 1929 are well reflected by the above-mentioned changes in the inspector's equipment. Here it seems appropriate to emphasize that in the development of the procedures to be followed by the inspectors, the CRL has always had the benefit of advice and instructions from the various laboratories in the Bureau, particularly in the verification of testing machines.

## SUMMARY OF INSPECTION OF LABORATORIES DURING TOURS 1 TO 10

*Number and Interests of Participating Laboratories.* — The laboratories that have availed themselves of this inspection service have been widely scattered, the CRL inspections having extended into all of the states and into Canada and Puerto Rico. Many laboratories have regularly requested inspections, thereby showing their continued interest in the work. Table I sets forth the number of laboratories involved in each of the tours, as well as the classification of the laboratories according to the interests represented. About 250 laboratories per tour have been inspected.

TABLE I.—CLASSIFICATION OF PARTICIPANT LABORATORIES IN INSPECTION TOURS 1 TO 10.

| Laboratory Classification | Tour 1 | Tour 2 | Tour 3 | Tour 4 | Tour 5 | Tour 6 | Tour 7 | Tour 8 | Tour 9 | Tour 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| Cement producer | 99 | 108 | 104 | 100 | 120 | 128 | 128 | 119 | 129 | 140 |
| Commercial testing | 45 | 48 | 30 | 37 | 66 | 73 | 50 | 41 | 36 | 39 |
| Highway department | 25 | 65 | 53 | 61 | 63 | 59 | 52 | 48 | 51 | 48 |
| School | 21 | 27 | 20 | 13 | 23 | 16 | 13 | 6 | 9 | 9 |
| Municipal | 11 | 10 | 9 | 6 | 10 | 10 | 7 | 4 | 7 | 4 |
| Federal | ... | ... | ... | ... | ... | 11 | 10 | 9 | 10 | 13 |
| Miscellaneous | 7 | 7 | 8 | 12 | 14 | 6 | 5 | 3 | 5 | 8 |
| Total laboratories | 208 | 265 | 224 | 229 | 296 | 303 | 265 | 230 | 247 | 261 |
| Approximate tour period | 1929–1930 | 1931–1932 | 1932–1933 | 1934–1935 | 1936–1938 | 1938–1940 | 1940–1943 | 1943–1946 | 1947–1950 | 1950–1954 |

*Effects of Inspection of Test Methods.*—It is difficult to measure closely the benefit of the demonstrations of test methods. However, in the early years of the work there was made a lengthy study of the apparent effects of the periodic inspections of test methods during the first four tours. Under the principal test methods there were listed numerous related details. Opposite each detail were shown the percentage of laboratories that had demonstrated that portion of the test in accordance with specification requirements in the respective tours. These percentages were separated for laboratories that had not been previously inspected and for those that had participated in at least one earlier inspection (**9**). A detailed report of that comparative study, presented in both tabular and graphic form, was contained in an Appendix to the 1935 Annual Report of Committee C-1 (**7**). Progressive improvement during the period of study was apparent in nearly all cases. Here it is to be noted that the tests covered by the 1935 study were fewer than the currently required tests, and in general were quite simple.

*Benefits of Inspection of Apparatus.*—The benefit of periodic inspection of apparatus has often been apparent (**7, 9**) in the course of the observations in the laboratories, also in related correspondence and discussions. Table I of the committee report just mentioned (**7**) in the discussion of methods illustrates the improvement in the acceptability of apparatus during the first four tours. There were some changes in tolerances during the interval covered by that table, including some appropriate lightening of tolerances in dimensions of used briquet molds, but in general the table illustrates the status of apparatus in those early tours, and indicates the benefit of periodic inspections. No very recent similar comparison has been made, but there are many reasons for believing that periodic reminders are still helpful in promoting proper maintenance of the more numerous and elaborate equipment of the present-day cement laboratory.

*Quantity of Apparatus Inspected in a Tour.*—The volume of work involved in an inspection tour is approximately indicated by the amount of equipment tested. Table II shows the quantities of the various apparatus that were inspected in each of Tours 1, 4, 7, and 10. The totals for those tours are regarded as quite typical for their respective times, and show the effects of the occasional changes in standard specifications for cement. For example, not long after the No. 200 sieve fineness test was omitted from the specifications, there was introduced the Wagner turbidimeter fineness apparatus, with the accompanying need for checking microammeters. Much later there appears in Table II the Blaine air-permeability fineness apparatus. The autoclave replaced the earlier steam chest in the soundness test, and equipment was provided to enable the inspectors to test the pressure gages. In due time there were involved details of flow tables, air-entrainment measuring equipment, and mechanical mixers.

TABLE II.—QUANTITIES OF APPARATUS TESTED IN CERTAIN INSPECTION TOURS.

| Apparatus | Tour 1 | Tour 3 | Tour 7 | Tour 10 |
|---|---|---|---|---|
| Steam chests | 201 | 211 | 278 | . . . |
| Autoclaves | . . . | . . . | 182 | 246 |
| Tensile testing machines | 217 | 223 | 308 | 237 |
| Compression machines | . . . | . . . | 230 | 341 |
| Fineness balances | 222 | 213 | . . . | . . . |
| Fineness weights | 1553 | 2753 | . . . | . . . |
| Wagner turbidimeters | . . . | . . . | 214 | 206 |
| Microammeters | . . . | . . . | 282 | 290 |
| Blaine air meters | . . . | . . . | . . . | 187 |
| Vicat apparatus | 251 | 265 | 368 | 312 |
| Gillmore needles (sets) | 262 | 269 | 299 | 300 |
| Burmister flow troughs | . . . | . . . | . . . | 181 |
| Flow tables | . . . | . . . | 176 | 245 |
| Briquet molds | 7764 | 9188 | 9584 | 5842 |
| Cube molds | . . . | . . . | 2440 | 3575 |
| Bar molds | . . . | . . . | . . . | 880 |
| Balances for mixes | 266 | 282 | 362 | 350 |
| Weights for mixes | 2951 | 3053 | 3330 | 3852 |
| Glass graduates | 477 | 580 | 681 | 629 |
| Cylindrical measures | . . . | . . . | . . . | 172 |
| Water retention apparatus | . . . | . . . | . . . | 108 |
| Autoclave pressure gages | . . . | . . . | . . . | 267 |
| Turbidimeter gages | . . . | . . . | . . . | 229 |
| Number of laboratories participating in tour | 208 | 224 | 265 | 261 |

When compressive strength of standard mortars entered into cement specifications, there arose the need for the verification of compression testing machines, and the related inspection of the numerous details of molds and bearing blocks. Proving rings were first added to the field equipment about 1940, permitting verification of the testing machines up to a load of 100,000 lb. Still later there were added proving rings of 200,000-lb capacity.

## COMPARATIVE TEST SAMPLES

*Purpose.*—For many years comparative tests of special lots of cement have been employed within various groups of participants, among whom were cement mills, commercial laboratories, Government agencies, highway departments, and others. Such tests were intended to promote improvement and uniformity in testing procedures and to afford an opportunity for the individual laboratories to compare results. Cooperative tests of cement were early employed in some of the work of Committee C-1, and when the work of the CRL was outlined it was provided that the laboratory should regularly prepare and distribute samples of cement for comparative test purposes (1).

*CRL Comparative Test Samples.*—In 1936, the CRL distributed the first of its samples of cement for general comparative test purposes. Tables III and IV briefly describe the samples distributed up to 1955, and indicate the scope of the work on each sample. Cement suitable for a planned comparative test sample is secured through appropriate arrangements, and is very carefully blended and packed for distribution. Proper announcements precede the preparation of the special lot of cement, samples of which are finally sent to each of the laboratories that request them. The results of all of the tests are summarized in detail in reports that are sent to each of the participants. The identity of the cement is not published. In the reports each individual laboratory is designated by a number that is revealed only to that laboratory.

TABLE III.—CRL COMPARATIVE TEST SAMPLES NOS. 1 TO 9. THE NUMBER AND CLASSIFICATION OF THE PARTICIPATING LABORATORIES.

| Comparative Sample | No. 1 | No. 2 | No. 3 | Nos. 4, 5 | Nos. 6, 7, 8 | No. 9 |
|---|---|---|---|---|---|---|
| Sample Distributed<br>Date of Report of Results | 1936<br>2-1-37 | 1938<br>1-6-39 | 1939<br>1-27-40 | 1940<br>3-3-40 | 1954<br>4-19-54 | 1955<br>3-15-56 |
| Participating Laboratories: | | | | | | |
| Cement producer | 24 | 66 | 106 | 114 | 140 | 136 |
| Highway | 12 | 41 | 36 | 27 | 29 | 33 |
| Commercial | 6 | 28 | 38 | 28 | 17 | 17 |
| Federal | 1 | 8 | 11 | 8 | 11 | 12 |
| Municipal | 1 | 7 | 3 | 4 | 3 | 7 |
| School | ... | 6 | 8 | ... | 2 | 4 |
| Miscellaneous | 1 | 5 | 6 | 7 | 4 | 4 |
| Total laboratories | 45 | 161 | 208 | 188 | 206 | 213 |

TABLE IV.—SCOPE OF TESTS ON NINE COMPARATIVE TEST SAMPLES.

| Sample | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 | No. 6 | No. 7 | No. 8 | No. 9 |
|---|---|---|---|---|---|---|---|---|---|
| Tests Involved: | | | | | | | | | |
| No. 200 sieve | X | | | | | | | | |
| Wagner turbidimeter | X | X | X | | | X | | X | X |
| Blaine meter | | | | | | X | | X | X |
| Tensile strength | X | X | X | | | X | | | X |
| Compressive strength | | X | X | | | X | | | X |
| Air entrainment | | | | | | X | | | X |
| Normal consistency | X | X | X | | | X | | | X |
| Setting time | X | X | X | | | X | | | X |
| Autoclave | | X | X | | | X | | | X |
| Chemical analysis | | X | X | X | X | | X | | |
| Flame photometer | | | | | | | X | | |
| Heat of hydration | | | X | | | X | | | |

The continued participation by so many laboratories in these comparative tests seems to indicate that the comparative samples are welcome. Also, the data and the related notes and correspondence have often been of great interest to Committee C-1 subcommittees engaged in the development of test methods (11). In the course of the work on samples 4 and 5 there were collected considerable data on analytical weights and balances. Those data were of interest not only to Committee C-1 in its work of that time but also aided one of the large Government bureaus in developing specifications for analytical balances for a much different line of work.

### TESTS OF ADDITIONS TO PORTLAND CEMENT

*Reasons for Tests.*—In Committee C-1, in the early discussions of "additions" to cement, there arose the question as to how there would be determined the acceptability of materials that might be proposed, from time to time, for intergrinding in very small amounts with the clinker in the manufacture of portland cement. There finally resulted the provision that an addition used under the existing specifications for portland cement must have been declared by Committee C-1 to be "non-harmful," (or "acceptable" under the then specifications for air-entraining cement) based on the results of tests carried out or reviewed by Committee C-1.

*Scope of CRL Tests of Additions.*—The CRL made three special series of tests on additions at the desire of Committee C-1. The programs of the work were developed in cooperation with the Working Committee on Additions, a subcommittee of Committee C-1. The various lots of cement were sampled during their grinding, with and without the proposed addition, at the various cooperating mills. Standard and special determinations, both physical and chemical, were made. Strength studies extended over a period of one year, and included not only small standard mortar specimens, but also beams and cylinders made of concrete of more than one proportion of mix. Observations were also made on length changes of bars under different storage conditions, also their durability in accelerated exposure conditions. The CRL arranged for personnel and equipment, performed the work, and prepared the final reports for submission to the Working Committee on Additions. The three series of tests of additions covered materials proposed for use as grinding aids, as follows:

1. "TDA," a material made by the Dewey & Almy Chemical Co., and proposed as a grinding aid in the manufacture of high-early-strength portland cement.
2. "TDA" for use as a grinding aid in other portland cement than high-early-strength.
3. "109-B," a material marketed by the Master Builders Co. as a grinding aid in the manufacture of the different types of portland cement.

The laboratory collaborated closely with the Subcommittee on Additions in the considerations of certain air-entraining additions that had been submitted to Committee C-1 and in the subsequent preparation of tentative specifications for air-entraining additions for use in the manufacture of air-entraining portland cement. Also, the CRL handled numerous inquiries from parties interested in learning about the conditions governing the acceptability of additions for intergrinding with the clinker.

### COOPERATION WITH SUBCOMMITTEES OF COMMITTEE C-1

The CRL has always cooperated with subcommittees of Committee C-1, numerous questions arising in the course of CRL work having been referred to the appropriate subcommittees. The laboratory has participated in numerous cooperative test programs of the subcommittees, and has been represented in the membership of some of the subcommittees. Among important features of such cooperative work were:

Assisting in the development of the compressive strength test method and in selection of requirements for equipment.

Participation in studies of methods of test for air-entrainment. The CRL staff was represented on the related subcommittee.

Special tests of standard sand.

Aiding in the cooperative studies of fineness tests.

Assembling information on design and performance of autoclaves throughout the country for the information of the Working Committee on Volume Change in its revision of test methods.

Collaboration with the Working Committee on Additions in the special tests made by the CRL as already described above.

*Coordination of Method of Test.*—In 1946, Committee C-1 established the Working Committee on Coordination of Methods of Test, designed to promote as much as feasible the uniformity of apparatus and methods in Committee C-1. The subcommittee is composed of the chairmen of the various Working Committees within Committee C-1. Ever since the subcommittee was formed, the Head of the CRL has been chairman of this coordinating subcommittee, which has been a very appropriate means of interchanging information and attaining agreement on apparatus and methods that may be of common interest to a number of the C-1 subcommittees. Information from the various subcommittees has been helpfully augmented by data collected in the CRL inspection work, as in the standardization of specifications for flow tables and mechanical mixers.

## SECRETARIAL AND EDITORIAL WORK

The CRL work includes the general correspondence and reports incidental to the laboratory's inspection service and comparative test programs. The correspondence has included numerous inquiries about apparatus and methods.

*Committee C-1.*—In his capacity as the Technical Assistant to Committee C-1, the Head of the CRL handles most of the numerous letters relating to the work of Committee C-1, assists in arranging agenda and numerous details preliminary to the meetings of the committee and its subcommittees, prepares and distributes the minutes and related ballot forms, and assembles the results of ballots. He also aids in editing and proof reading the committee's various recommendations to the Society, and in assembling the various cement specifications and test methods into the compilation known as "ASTM Standards on Cement." This book is sponsored by Committee C-1 and is reprinted when the total changes and revisions warrant it, usually at about 2-year intervals.

*Manual on Cement Testing.*—About the time the CRL was being established, a special subcommittee in Committee C-1 had completed a helpful compilation guide, with information, to supplement the ASTM cement test methods. Since the publication of the book "AS TM Standards on Cement," the Manual has formed an Appendix thereto. Aided by the appropriate C-1 subcommittees of Committee C-1, the CRL has regularly provided the periodic editing to keep the Manual in line with currently applicable standards and knowledge.

## SPECIAL STUDIES OF APPARATUS AND METHODS

The CRL was not intended to be a research laboratory, but as time permitted there were to be occasional studies of some of the questions and data arising in its field of operations (1). The time devoted to such study was not continuous, because efforts were made to maintain the laboratory inspection service at a suitable rate. Some of the studies were only extensive enough to answer pertinent current questions. The more important of these studies, arranged in somewhat chronological order and with the approximate year of the work indicated, were as follows:

*Film.*—The CRL prepared a 16-mm film illustrating the proper method of performing the tests then required by standard specifications. This film was used in some of the early talks by the inspectors, and a copy was loaned on several occasions to interested laboratories. (Work done in 1930.)

*Early Inspection Data.*—The results of laboratory inspections of the first tour were summarized for the information of the subcommittees of Committee C-1 (4). The elaboration of this summary also served as the basis for a paper (5) presented by P. H. Bates and J. R. Dwyer at the 1930 Annual Meeting of the Portland Cement Assn. (1930–1931).

*Sub-Sieve Fineness.*—Acting on the advice of P. H. Bates, the then pertinent subject of measurement of sub-sieve fineness of cement was investigated by L. A. Wagner who was then a CRL staff member. Following trials of a number of turbidimetric devices, Wagner devised the turbidimeter that bears his name (6). This turbidimeter was quickly used by the Bureau of Reclamation in the cement specifications for Hoover Dam. The work included the design of air separator stacks and their method of use to secure suitably-clean fractions for the microscopic measurements (1931–1933).

*Calibration of Turbidimeter.*—A large group of laboratories cooperated with L. A. Wagner in the studies that led to a satisfactory basis for calibration of the Wagner turbidimeter, and the calibration sample thus evolved became the fore-runner of the current NBS Standard Sample No. 114. With suggestions from Bureau personnel, Wagner devised an appropriate timing buret to be used with the turbidimeter (1933–1934)

*Moist Cabinet.*—Based on experience in the inspection work, the CRL designed a temperature-controlled moist cabinet for the Bureau cement laboratory. Built of transite by the Bureau, the cabinet was recently retired after about 20 years service in the Bureau cement laboratory (1935).

*Effects of Inspections.*—Study was made of the inspectors' findings during the inspection of apparatus and methods during the first four tours to determine what might be the benefits of the work as evidenced in repeated inspections (7). The results of the study were incorporated in tables that formed part of an ASTM paper by Dwyer (9) (1935–1937).

*Planeness of Bearing Surfaces.*—Questions relating to requirements for planeness of bearing surfaces used in compression tests of standard mortar cubes led to an investigation of the effects of different degrees of departure from planeness. Numerous cubes were tested at different ages, using steel bearing blocks that had been ground in the Bureau optical shop to secure certain different degrees of concavity and convexity. The results, supporting the present standard tolerances, were presented by J. R. Dwyer in an ASTM paper (8) (1936).

*Turbidimeter Stirrer: Turbidimeter Tests of Fine Sands.*—John R. Gran devised a stirrer and guard that could be used in lieu of the somewhat variable brush in preparing the suspension for the Wagner turbidimeter test. The stirrer was successfully used by a number of laboratories. The standard method now permits the use of any stirring device that will be equally efficient as the brush as measured by determinations on a standard sample. Gran also made some studies of the application of the Wagner turbidimeter to the tests of fine sands and reported his results in a paper in the ASTM BULLETIN (10).

*Speed of Testing.*—The CRL participated in some of the studies related to the development of the standard test for compressive strength of mortar cubes. As part of that work, a series of tests was made by H. E. Newcomer to determine what effect on the observed strength might result from variations in the rate of application of load to the 2-in. cubes. The unpublished report was made available to the Subcommittee on Strength (1938).

*Sieve Tests of Standard Sand.*—The laboratory collaborated with Subcommittee III (Fineness) in some special sieve tests of certain lots of standard 20–30 sand. The tests involved a number of laboratories and four certified standard sieves. The CRL secured for the subcommittee the results of the sieve mesh measurements made by the Bureau in the original certification tests of the sieves. The subsequent recommendations of the subcommittee led to the revised 20–30 sand requirements as still in effect (1929).

*Tests of Air-Entraining Cements.*—Because of the then live interest in the early development of appropriate apparatus and methods for testing air-entraining cements, two members of the CRL staff (J. C. Yates and J. R. Dwyer) collaborated with R. L. Blaine of the Bureau staff in a study of air-entraining cements. (Mr. Blaine is now Chief of the Section in which the CRL has always been located.) The study included the tests of mortars and concretes made from a large number of cements containing an air-entraining agent added by the then mill practice. The results of those tests were reported in an ASTM paper (12) which included data on the relative performance of some of the apparatus and methods employed in the tests of such cements (1944). In subsequent years, the CRL continued its interest in air-entrainment measurements and for a long time has been represented on the Working Committee on Methods of Test for Air-Entrainment, participating in some of the subcommittee's test programs.

*Autoclave Data.*—Because of some important questions that had arisen, the CRL distributed a questionnaire concerning autoclaves—their design details, dimensions, heating and cooling performance, and practice concerning use of thermometers. A digest of some of the resulting information was given to the C-1 Working Committee on Volume Change and Soundness of Portland Cement (1941–1942).

*Strength Test Apparatus.*—On request, the CRL prepared and submitted to the Sponsoring Committee on Portland Cement a summary of certain details of compression testing equipment observed by the inspectors during their visits to the various cement laboratories (1944).

*Flow Table Calibration Sample.*—In the course of flow table inspections, the need for a suitable calibration material became more pressing. Tests had been made with ground limestone prepared by an industry research laboratory. Also, there was investigated the possibility of using a mixture of ground silica and kerosine as had been tried in another ASTM Committee years ago. However, more satisfactory results were desired, and trials were made of various ground materials mixed with oil (13). After numerous tests of commercially available materials, the CRL ground some standard Ottawa sand to the fineness required, as determined by trials of the ground sand mixed with a mineral oil suggested by Mr. R. C. Hardy of the Rheology and Lubrication Section in the Bureau. The results were very encouraging. Additional lots of sand were ground and portions were sent to interested laboratories, upon request, for incorporation with their locally-purchased oils (1945–1949). Later, the dry material was distributed together with a bottle of oil from a given known lot of oil. Still later the method of grinding the sand was improved, a lined mill and pebbles being used. The instructions for use were bettered, as the result of experience, and a glass container was required for mixing and storage. The new calibration sample has been in steady demand and has been very helpful in checking the performance of flow tables, particularly in the case of newly purchased or recently remounted tables (1952–1955).

*Specifications for Flow Table.*—The CRL inspections of flow tables, plus the use of the calibrating sample, resulted in the accumulation of much related information. In addition, for some months the CRL had carried into the field a portable flow table, mounted on a very heavy metal base. Tests made with the calibration material, using both the CRL portable flow table and the table of the inspected laboratory, served well to show the need for proper design of the table and its mounting. The accumulation of flow table data proved helpful in the later discussions that led to the adoption of flow table specifications as recommended through the Subcommittee on Coordination of Methods of Test (1945–1949).

*Mechanical Mixing of Mortars.*—In the development of mechanical mixing of standard test mortars, various subcommittees of Committee C-1 cooperated in the studies that led to the method that was finally recommended to Committee C-1 through the Working Committee on Coordination of Methods of Test. During their inspections of many cement laboratories, the CRL inspectors collected information and suggestions that were helpful in the preparation of the new test method and in the specifications for the mixer (1951–1953).

## MISCELLANEOUS OTHER ACTIVITIES

The CRL work has included additional miscellaneous activities, some of which are briefly mentioned here to convey an idea of their nature.

The CRL staff has furnished a Committee C-1 representative on ASTM Committee C-9 on Concrete and Concrete Aggregates, also on five subcommittees of ASTM Committee E-1 on Methods of Testing, the titles of those subcommittees being:

Calibration of Mechanical Testing Machines and Apparatus.
Effect of Speed in Mechanical Testing.
Conditioning and Weathering.
Ceramic Laboratory Apparatus.
Metalware Laboratory Apparatus.

Occasional special reports on CRL work were presented to Committee C-1 by CRL staff members, including Wagner, Newcomer, Giles, Dise, Schwartz, Benedict, Dwyer.

Paper by L. A. Wagner at State College, Pennsylvania.

Remarks on importance of correct procedure in compressive strength testing of concrete, presented by Dwyer at a meeting of the National Ready Mixed Concrete Association and the National Sand and Gravel Association.

Brief discussions before AASHO Committee on Materials, at that committee's invitation, the remarks relating to matters of current common interest to the committee and the CRL.

Occasional brief talks before school materials classes, concrete conferences, Bureau groups.

Cooperation with the Bureau and the ASTM in the advance review of numerous proposed papers prior to acceptance for publication.

### CRL Personnel

The staff of the CRL has included numerous assistants. Some of the assistants remained for only a short time, while others stayed for a considerable number of years. A few of the staff members worked only in Washington, as in the case of special tests of additions to cement, which tests were made for Committee C-1. The names of CRL assistants to date are here listed, as follows:

Benedict, S. W.
Burdick, M. D.
Casanova, A. Y., III
Clemmer, S. R.
Curry, R. L.
DeFore, M. R.
Dise, J. R.
Giles, J. H., Jr.
Gran, J. R.
Haverfield, J. W.
Hicks, T. D.
Kelly, T. M.
Kohli, C. B.
Kurtz, H. J.
Messimer, L. A.
Middleton, W. W.
Milmore, O.
Moffitt, R. S.
Myers, M. E.
Newcomer, H. E.
Schwartz, J. I.
Siegle, A. G.
Thibodeau, W. E.
Tomes, L. A.
Wagner, L. A.
Werner, G. H.
Yates, J. C.
Yourshaw, A. M.

Acknowledgment is made to all of the above named past and present CRL assistants for their help. Their earnest efforts in the laboratory and their generous devotion to the exacting demands of travel schedules have made the CRL work possible. Particular mention is made of the efforts of L. A. Wagner and the late H. E. Newcomer. They were the first two assistants, and they ventured into the field in the commencement and development of the inspection procedure. Mr. Wagner remained with the CRL for about seven years; and Mr. Newcomer stayed for twelve years until his promotion to other work in the Bureau. L. A. Messimer joined the CRL in its early years and remained for almost ten years until he transferred to the Bureau staff on other work in 1941.

Also among the assistants who stayed with the CRL for approximately four years or longer were: S. W. Benedict, R. L. Curry, M. R. DeFore, J. R. Gran, T. M. Kelly, J. C. Yates. More recently, the CRL is indebted to J. R. Dise for his diligent attention to the maintenance and improvement of many details of the field work. Mr. Dise joined the CRL in early 1949. On October 4, 1955, he succeeded John R. Dwyer as Head of the CRL, and also as the Technical Assistant to Committee C-1.

### *Acknowledgments:*

It seems fitting that this review of CRL activities should be supplemented by some expression of appreciation for the assistance received by the laboratory. It would indeed be difficult to prepare an adequate list. In the early part of this report, tribute was paid to those who had planned, sponsored and supported the project. During the subsequent years, assistance and encouragement were received from many sources—officials and laboratories of Government bureaus and the Portland Cement Assn., from the ASTM, and from the many cement laboratories of various interests throughout the country. To all of the laboratory's many friends throughout the formative and the later years, there are offered sincere thanks on behalf of the CRL.

### Former Members of the Subcommittee on Cement Reference Laboratory

In the preparation of this report, interest was attracted to the list of names of former members of the CRL Subcommittee. Necessarily, for numerous reasons, there were occasional changes in the personnel of this subcommittee that was established in 1929 to aid and guide the CRL. It now seems appropriate to record the names of the former members in tribute to their help to the subcommittee. The former members were:

Ashton, Ernest
Chapman, C. M.
Conn, C. F. (Chairman 1932–35)
Cook, H. K.
Goldbeck, A. T.
Greenman, R. S.
Insley, Herbert
Jackson, F. H. (Vice-Chairman 1954)
Kelley, F. W. (Chairman 1929–32)
Lindsay, G. L.
Mattimore, H. S.
Moore, O. L.
Walter, L. W.
Warren, G. E. (Chairman 1935–54)

In connection with the foregoing list, it should be mentioned that Mr. Jackson served for more than 25 years as a member of the subcommittee, Mr. Mattimore for more than 20 years. Also, George Warren served for almost 20 years as a very helpful and encouraging chairman of the subcommittee that was formed to guide the project that he had once helped to establish.

Report submitted on behalf of the Subcommittee on Cement Reference Laboratory,

D. S. MACBRIDE,
*Chairman.*

SUBCOMMITTEE MEMBERS

HAROLD ALLEN
A. A. BATES
R. L. BLAINE
C. L. DAVIS
W. C. HANNA
H. F. HEDDERICH
R. R. LITEHISER
G. L. OTTERSON
F. V. REAGEL
BAILEY TREMPER
STANTON WALKER

## REFERENCES

(1) Report of Committee C-1 on Cement for 1929. *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part I, pp. 262–265 (1929). Contains report of the Special Committee on Cement Reference Laboratory.

(2) *Technical News Bulletin*, National Bureau of Standards, March, 1930, p. 7. Article refers to current need for and study of tolerances for cement testing apparatus.

(3) *Technical News Bulletin*, National Bureau of Standards, No. 156, Apr., 1930, p. 35. Concerns early progress of work.

(4) Report of Committee C-1 on Cement for 1930. *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 426 (1930). Appendix contains report of CRL Subcommittee and covers work at 119 laboratories.

(5) J. R. Dwyer and P. H. Bates, "The Cement Reference Laboratory at the Bureau of Standards. Its Origin, Purpose, and a Survey of Its First Year's Work." Paper presented at the Annual Meeting of the Portland Cement Assn., Nov. 19, 1930.

(6) L. A. Wagner, "Rapid Method for Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

(7) Report of Committee C-1 for 1935, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 238 (1935). Appended is report of Subcommittee on Cement Reference Laboratory, containing a comparison of apparatus and methods as observed in first four inspection tours.

(8) J. R. Dwyer, "Effect of Departure from Planeness of Bearing Surfaces on Compressive Strength of 2-in. Mortar Cubes," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 351 (1936).

(9) J. R. Dwyer, "The Cement Reference Laboratory," ASTM BULLETIN, No. 87, Aug., 1937, p. 12.

(10) J. R. Gran, "Some Suggested Practice in Estimation of Particle Size Gradation of Granular Materials," ASTM BULLETIN, No. 88, Oct., 1937, p. 17.

(11) Report of Committee C-1 for 1939. *Proceedings*, Am. Soc. Testing Mats., Vol. 39, pp. 321–332 (1939). The appended report of the Subcommittee on Cement Reference Laboratory summarizes, in tabular and graphical form, the results of tests on CRL Comparative Test Sample No. 2.

(12) R. L. Blaine, J. C. Yates, and J. R. Dwyer, "Testing of Portland Cements Containing Interground Vinsol Resin," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 732 (1945).

(13) Report of Committee C-1 for 1948. *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 247 (1948). Contains brief account of study of flow table calibration problem.

# REPORT OF COMMITTEE C-2

ON

# MAGNESIUM OXYCHLORIDE AND MAGNESIUM OXYSULFATE CEMENTS*

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements met once during the year: on June 21, 1956, in Atlantic City, N. J., during the Annual Meeting of the Society.

The committee consists of 19 members, of whom 7 are classified as producers, 4 as consumers and 8 as general interest members.

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee C-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Tentative Method of Test for:*

Yield of Magnesium Oxychloride Cement (C 389 - 56 T), and

*Tentative Field Test for:*

Yield of Magnesium Oxychloride Cement (C 388 - 56 T).

These recommendations were accepted by the administrative committee on November 15, 1956, and the new methods appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

## ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

Committee C-2 recommends that the Tentative Methods of Test for Ignition Loss and Active Calcium Oxide in Magnesium Oxide for Use in Magnesium Oxychloride Cements (**C 247 - 54 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

This recommendation has been submitted to letter ballot of the Committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 19 members; 16 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. S. NEWMAN,
*Chairman.*

K. M. BERG,
*Secretary.*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE C-3
## ON
# CHEMICAL-RESISTANT MORTARS*

Committee C-3 on Chemical-Resistant Mortars held three meetings during the year: on June 18, 1956, in Atlantic City, N. J., on November 26 and 27, 1956, in Cleveland, Ohio, and on February 19, 1957, in New York City.

The committee consists of 31 members, of whom 13 are classified as producers, 8 as consumers, 8 as general interest members, and 2 as consulting members.

### NEW TENTATIVES

The committee recommends for publication as tentative the following as appended hereto:[1]

*Specification for:*

Resin-Type Chemical-Resistant Mortars,

*Method of Test for:*

Compressive Strength of Chemically Setting Silicate-Type Chemical-Resistant Mortars,

*Recommended Practice for:*

Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars,
Use of Hydraulic Cement Mortars, and
Use of Resin-Type Chemical-Resistant Mortars.

### REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Bond Strength of Chemical Resistant Mortars (**C 321 – 56**)[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

*Section* 5.—Add the following footnote to this section:

> [1] Brick to be used with hydraulic cements shall not be dried.

*Section* 8 (*a*).—In the second line following the word "sulfur" add "and hydraulic cement." At the end of this section, add a sentence "Specimens made with hydraulic cement mortars shall be stored for 14 days in a moist cabinet at 23 ± 2.2 C (73.4 ± 4 F) and at a relative humidity of not less than 90 per cent."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

### SUBCOMMITTEE ACTIVITIES

*Subcommittee T-2 on Physical Properties* (R. S. Mercer, chairman) is preparing test methods for absorption, porosity, and coefficient of thermal expansion of mortars.

*Subcommittee T-3 on Chemical Properties* (W. A. Meeley, chairman) is considering methods of testing chemical resistance of mortars by weight change, compressive strength, and appearance of both the exposed specimens and the test solutions.

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*** Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.**

[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] **1955 Book of ASTM Standards, Part 3.**

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 31 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

BEAUMONT THOMAS,
*Chairman.*

E. A. REINECK,
*Secretary.*

# REPORT OF COMMITTEE C-7
## ON
## LIME*

Committee C-7 on Lime held two meetings during the year: on June 22, 1956, in Atlantic City, N. J., during the Annual Meeting of the Society, and on February 5, 1957, in Philadelphia, Pa., during ASTM Committee Week.

The committee records with regret the death of F. O. Anderegg, a member of the committee since 1932.

R. K. Thomas has succeeded D. G. Hamme as chairman of Subcommittee V.

The committee consists of 48 members, of whom 22 are classified as producers, 5 as consumers, 21 as general interest, and 2 honorary members.

A special committee prepared extensive revisions of the Regulations Governing Committee C-7 which have been approved by the main committee.

### NEW TENTATIVE

The committee recommends for publication as tentative the proposed Method of Testing Quicklime and Hydrated Lime for Neutralization of Waste Acid, as appended hereto.[1]

### REVISION OF TENTATIVE

The committee, jointly with Committee C-11 on Gypsum, recommends the following revisions of the Tentative Specification for Inorganic Aggregates for Use in Interior Plaster (**C 35 – 54 T**):[2]

*Section 3(a).* — Change the third, fourth, and fifth values for perlite by volume, max, from "45, 85, and 95" to read "60, 95 and 98."

*Section 4(a).*—Change "7½" to read "6."

### REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

**Standard Specifications for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 – 42):**[2] Revise as appended hereto.[3]

**Standard Methods of Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (C 50 – 27):**[2] Revise as appended hereto.[3]

**Standard Methods of Physical Testing of Quicklime and Hydrated Lime (C 110 – 49):**[2]

*Section 7(a).*—Change the second sentence to read as follows:

> A ring mold such as described in Section 5(*b*) shall be lubricated with a thin film of water, placed on a porcelain base plate (see Sections 9 and 10) or a disposable plaster base plate (see Section 10), filled with the paste which has been adjusted to standard consistency as described in Section 6, and struck off level.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

[3] The revised standards appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

*Section 10(a)*.—Replace the second sentence with the following:

> Before making the determination, the porcelain plates shall be dried overnight in an oven at temperatures of between 212 and 230 F (100 and 110 C), and permitted to cool to room temperature. The plaster plates shall be dried overnight over calcium chloride at room temperature.

### WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Specifications for Hydrated Lime for Varnish Manufacture (**C 47 - 27**).[2] A survey indicated that this specification is not used by the varnish manufacturers and there is no commercial interest in such a specification.

### EDITORIAL CHANGE

The committee recommends editorial changes as follows in the Tentative Specifications for Quicklime and Hydrated Lime for Water Treatment (**C 53 - 52 T**):[2]

*Section 1.*—Change the first sentence of this section to read, "These specifications cover lime suitable for all water-treating applications."

*Section 3(b)*.—Change Note 2 to read as follows:

> NOTE 2.—For the majority of water-treating applications, high-calcium lime is preferred. However, for silica removal from boiler feedwater, dolomitic lime should be employed.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

### SUBCOMMITTEE ACTIVITIES

*Subcommittee II on Structural Lime* (B. L. Corson, chairman) will make further studies on the effect of unhydrated magnesia in lime used in mortars relative to compressive strengths of mortars and bond strengths, and the use of a screen for supporting a pat of lime instead of the ceramic discs used in the plasticity test. The subject of efflorescence will be considered.

*Subcommittee VIII on Pozzolans* (John L. Minnick, chairman) is considering carrying out cooperative tests on fly ash and natural pozzolanic materials.

This report has been submitted to letter ballot of the committee, which consists of 48 members; 34 members returned their ballots, of whom 33 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. A. MURRAY,
*Chairman.*

L. E. JOHNSON,
*Secretary.*

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE C-8

ON

# REFRACTORIES*

Committee C-8 on Refractories held two meetings during the year: the 90th meeting at White Sulphur Springs Hotel, Mann's Choice, Pa., on September 19, 1956, and the 91st meeting at the Benjamin Franklin Hotel, Philadelphia, Pa. on February 8, 1957.

The committee consists of 46 members, of whom 23 are classified as producers, 16 as consumers, and 7 as general interest members.

New members elected during the year were R. W. Limes, Allegheny Ludlum Steel Corp., J. M. Warde, Electro-Metallurgical Co. and T. E. Brady, Surface Combustion Corp.

The regulations governing Committee C-8 were revised to allow for the appointment of dual representation of corporate members.

## NEW TENTATIVE

The committee recommends for publication as tentative the Classification of Castable Refractories as appended hereto.[1]

## TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

### Method for Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 – 49):[2]

*Section 4.*—In the fourth sentence, replace "refractory kaolin" with "a mixture of 75 per cent plastic kaolin and 25 per cent of a highly calcined purified alumina, all of which passes a No. 100 ASTM sieve (equivalent to mesh No. 100 of the Tyler standard series)."

*Section 5.*—In the fifth sentence, change "20 F (10 C)" to read "7.5 F (4 C)."

*Section 6.*—In the fifth sentence, change "temperatures of 720, 260, and 180 F (400, 200, and 100 C)" to read "10 min intervals."

### Specifications for Fireclay-Base Castable Refractories for Boiler Furnaces and Incinerators (C 213 – 55):[2]

*Section 6.*—Change to read as follows:

6. *Sampling.*—Castable refractories shall be selected from each shipment of a carload or less as follows:

| Bags in Lots of | Number of Bag Samples |
|---|---|
| 100 or less | 2 |
| 100 to 300 | 3 |
| Over 300 | 5 |

### Method of Test for Disintegration of Refractories in an Atmosphere of Carbon Monoxide (C 288 – 56):[3]

*Section 3(c).*—Delete this section.

*New Section.*—Add the following new Section 4, on Unfired Samples, renumbering the present Section 4 and subsequent sections accordingly:

4. *Unfired Samples.*—Unfired refractories shall be prepared for testing by firing for a period of

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

[3] 1956 Supplement to Book of ASTM Standards, Part 3.

5 hr and under oxidizing conditions at one of the following temperatures:

(*a*) 1000 F, for the purpose of removing combined water.

(*b*) 2000 F, for the purpose of removing combined water and inhibiting compounds.

*Section 5.*—Renumber as Section 6 and change the first sentence to read, "Report any prefiring temperature of the specimens and the conditions of each specimen at each inspection describing the extent of any spotting and indicating the degree of disintegration, classified as follows:"

### Classification of Fireclay Refractory Brick (C 27 – 56):[3]

*Table I.*—For low-duty brick, change the P.C.E. min from "19" to read "15."

## REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Classification of Insulating Fire Brick (**C 155 – 47**)[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

*Table I.*—Change the bulk density for Group 26 brick from "52" to read "54" lb per cu ft.

## EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

### Suggested Practice for Use with ASTM Panel Spalling Tests for Refractories:[4]

Add the following as the sixth paragraph of the Section on Furnace Construction:

> As an aid to obtain more uniform temperature distribution from the bottom to the top of the face of the test panel, the distance between the outer surface of either bag wall and the opposite interior surface of the furnace wall may be maintained at $1\frac{1}{4}$ in.

### Specifications for Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens (C 63 – 51):[2]

*Section 8(c).*—In the first paragraph change "high-duty fireclay brick" to read "regular high-duty fireclay brick or semi-silica brick."

### Specifications for Refractories for Heavy Duty Stationary Boiler Service (C 64 – 51):[2]

*Section 6.*—In the first sentence of Paragraphs (*a*), (*c*), and (*e*), change "high duty fireclay brick" to read "regular high duty fireclay brick or semi-silica brick;" in the first sentence of paragraph (*b*) change "high-duty fireclay brick" to read "spall resistant high duty fireclay brick or semi-silica brick;" in the first sentence of paragraph (*d*) change "super duty fireclay brick" to read "spall resistant super duty fireclay brick."

### Specifications for Refractories for Incinerators (C 106 – 51):[2]

*Section 5(a).*—In the first paragraph change "high duty fireclay brick" to read "regular high duty fireclay brick."

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

*Tentative Method of Test for:*

Modulus of Rupture of Castable Refractories (**C 268 – 55 T**), and

Permanent Linear Change on Firing of Castable Refractories (**C 269 – 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

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[4] 1952 Manual of ASTM Standards on Refractory Materials, p. 144.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Industrial Survey* (R. P. Stevens, chairman) has supplied a new survey on heat treating furnaces. A revision of the survey of continuous bottle glass furnace is under way and three other surveys are scheduled for revision during the year.

*Subcommittee II on Research* (R. B. Sosman, chairman) is working on a statement of the problem of permeability of refractories to gases.

*Subcommittee III on Tests* (S. M. Phelps, chairman) has suggested a new procedure for the guidance of the Sections in submitting tentatives or standards for ballot.

*Section A on Load* (A. J. Metzger, chairman) is continuing the study of the shape factor in connection with modulus of rupture determinations and temperature gradients in various refractories when heated according to standard load test heating schedules. The section has been assigned the problem of recommending a load test for mullite and similar refractories.

*Section B on Spalling* (L. J. Trostel, chairman) prepared the revisions of Method C 38 - 49 referred to earlier in the report. An incomplete assignment consists of a thermal spalling test for blast furnace brick.

*Section C on Temperature* (J. L. Carruthers, chairman) is developing data from a comprehensive testing of various types of PCE furnaces.

*Section E on Chemical Analysis* (H. A. Heiligman, chairman).—The Bureau of Standards has completed analyses of two new standard samples of silica brick with nominal $Al_2O_3$ contents of 0.2 and 0.5 per cent. Analyses to be conducted in ten industrial laboratories before certificates are issued have been arranged by the section.

The Task Force on Hydration (G. R. Eusner, chairman) has nearly completed methods covering brick and dead-burned magnesite. Another year will be required to develop a suitable method for dead-burned dolomite.

*Section F on Refractory Insulation* (W. L. Stafford, chairman) is working on specifications for insulating fire brick for linings of industrial furnaces in which there are negligible atmosphere problems. The section desires more active participation of consumers in work on testing insulating firebrick under load in a reducing atmosphere at high temperature.

*Section G on Porosity and Permanent Volume Change* (L. C. Hewitt, chairman) has completed a method for the determination of porosity and bulk density of carbon and carbon-ceramic refractories and is now working on a method for determining the bulk specific gravity of granular materials by a mercury immersion method.

*Section H on Mortars, Plastics and Castables* (G. H. Anthony, chairman) recommends the continuation without change of the Tentative Method of Test for Modulus of Rupture of Castable Refractories (C 268 - 55 T) and Tentative Method of Test of Permanent Linear Change on Firing of Castable Refractories (C 269 - 55 T).

*Section J on Carbon Monoxide Disintegration* (K. A. Baab, chairman) prepared the revision of Method C 288 referred to earlier in the report.

*Subcommittee IV on Heat Transfer* (C. L. Norton, chairman) is continuing work on the determination of the thermal conductivity of castables. After its completion tests will be started on plastics.

*Subcommittee VI on Nomenclature* (A. W. Allen, chairman) is undertaking work on a list of additional standard terms relating to refractories, taken from the unofficial glossary.

*Subcommittee VII on Specifications* (J. D. Sullivan, chairman) prepared the editorial revisions of Specifications C 63, C 64, C 106, C-153 and the tentative revisions of Specification C-213 referred to earlier in the report.

*Subcommittee VII AD HOC on Pouring Pit Refractories* (J. A. Roslund, chairman) is working on a specification for pouring pit refractories.

*Subcommittee VIII on Editorial* (W. R. Kerr, chairman) is preparing a new edition of the Manual on Refractories.

*Subcommittee IX on Classifications* (J. A. Kayser, chairman) prepared the tentative revisions of the Classification of Fireclay Brick (C 27 – 55) and the new Tentative Classification of Castable Refractories referred to earlier in the report. Comments are being solicited from members of The Refractories Institute as well as all members of Committee C-8 on a proposed classification of silica brick.

*Subcommittee XI on Special Refractories* (A. Maupin, chairman) is continuing the study of a classification of mullite refractories. Several new methods are believed to be needed to determine certain properties of these refractories.

*Subcommittee XII on Carbon Refractories* (E. B. Snyder, chairman) has submitted a carbon refractories reheat test as a revision of Method C 113 – 46. Work is continuing on a permeability test for carbon refractories.

*Subcommittee XV on Basic Granular Refractories* (J. J. Hazel, chairman) plans to contact the Open Hearth Committee of the American Institute of Mining and Metallurgical Engineers for information on requirements for burnt dolomite for use in open-hearth furnaces. No conclusive information has yet been obtained.

This report has been submitted to letter ballot of the committee, which consists of 46 members; 46 members returned their ballot, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. HAZEL,
*Chairman.*

S. M. PHELPS,
*Vice-Chairman.*

L. J. TROSTEL,
*Secretary.*

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-8 presented to the Society through the Administrative Committee on Standards revisions of the Tentative Method of Test for Permanent Linear Change on Firing of Castable Refractories (C 269 – 55 T). The revisions were accepted by the Standards Committee on November 25, 1957, and the revised tentative method appears in the 1957 Supplement to Book of ASTM Standards, Part 3, and in the November, 1957 Compilation of ASTM Standards on Refractories.

# REPORT OF COMMITTEE C-9

ON

# CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held two meetings during the year: on June 19, 1956, during the Annual Meeting of the Society in Atlantic City, N. J., and on September 20, 1956, during the Second Pacific Area National Meeting of the Society in Los Angeles, Calif.

The committee records with sorrow the death of L. W. Walter, an honorary member, who had been a member of the committee since 1916.

At the meeting of June 19, 1956, H. J. Gilkey and A. T. Goldbeck were elected to Honorary Membership on the committee.

The Sanford E. Thompson Award, established by the committee in 1938, in honor of its first chairman, is made for a paper of outstanding merit on concrete or concrete aggregates presented before the Society. The award this year will be made to T. C. Powers of the Portland Cement Assn. for a paper entitled "Basic Considerations Pertaining to Freezing and Thawing Tests," which was presented at the 1955 Annual Meeting of the Society.

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee C-9 presented to the Society through the Administrative Committee on Standards the recommendation that the Specification for Packaged, Dry, Combined Materials for Mortar and Concrete be published as tentative. This recommendation was accepted by the Standards Committee on September, 5, 1956, and the tentative specification appears in the 1956 Supplement to Book of ASTM Standards, Part 3, bearing the designation C 387 – 56 T.

## NEW TENTATIVES

The committee recommends for publication as tentative the following methods and specifications as appended hereto:[1]

Specifications for Raw or Calcined Natural Pozzolans for Use as an Admixture in Portland Cement Concrete.

Method of Test for Measuring the Rate of Hardening of Mortars Wet-Sieved from Concrete Mixtures by Proctor Penetration Resistance Needles.

## REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

**Tentative Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland-Cement Concrete (C 311 – 54 T):**[2]

*Section 15.*—Change to read as follows:

15. (*a*) Thoroughly mix by grinding together, in a mortar or in a mechanical blender, fly ash and hydrated high-calcium lime ($Ca(OH)_2$) in a proportion of $2\frac{1}{2}$ parts of fly ash to 1 part of hydrated lime by weight. Add 7 g of this mixture to 10.0 ml of $H_2O$ in a small plastic vial of approximately 25-ml capacity, stirring until the mixture is uniform. Seal the vial and store at 38 C.

(*b*) Open the vial at the age of 28 days and transfer the contents to a 250-ml casserole.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] These new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

Break up and grind the cake with a pestle, adding a small amount of water, if necessary, so that a uniform slurry containing no lumps is obtained (Note 1). Add sufficient water to make the total volume 200 ml. Let stand 1 hr at room temperature with frequent stirring. Filter through a medium-texture filter paper into a 500-ml volumetric flask. Wash thoroughly with hot water (8 to 10 times).

NOTE 1.—At times it may be necessary to break the vial and peel off the plastic from the solid cake. In such cases, care should be exercised to avoid the loss of material and to remove all solid material from the fragments of the vial. If the cake is too hard to break up and grind in the casserole, a mortar should be used.

(*c*) Neutralize the filtrate with HCl (1:3), using 1 to 2 drops of phenolphthalein solution as the indicator. Add exactly 5 ml of HCl (1:3) in excess. Cool the solution to room temperature and fill the flask to the mark with distilled water. Determine the amount of sodium and potassium oxides in the solution, using the flame photometric procedure described in Sections 15 to 18 of the Tentative Methods of Chemical Analysis of Portland Cement (ASTM Designation: C 114 - 56 T), except that the standard solutions shall be made up to contain 8 ml of $CaCl_2$ stock solution per liter of standard solution, and the solution as prepared shall be used in lieu of the solution of cement (Note 2).

NOTE 2.—The standard solutions made up with 8 ml of $CaCl_2$ stock solution contain the equivalent of 504 ppm of CaO. Tests have shown that this amount closely approximates the amount of calcium dissolved in the test solution.

### Tentative Specifications for Fly Ash for Use as an Admixture in Portland-Cement Concrete (C 350 - 54 T):[2]

*Table 1.*—Change "Silicon dioxide ($SiO_2$), min, per cent...40.0" to read "Silicon dioxide ($SiO_2$) plus aluminum oxide ($Al_2O_3$) plus iron oxide ($Fe_2O_3$), min, per cent...70.0."

### Tentative Method of Test for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293 - 54 T):[2]

*Section 2.*—Add the following as a new second sentence: "The testing machine shall conform to the requirements of Section 15 of the Methods of Verification of Testing Machines (ASTM Designation: E 4)."

### Tentative Method of Test for Lightweight Pieces in Aggregate (C 123 - 53 T):[2]

*Section 3 (a).*—Add the following as a new second sentence, "Bromotrichloromethane may be used as a heavy liquid having a specific gravity of 2.00."

### Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 - 54 T):[2]

*Section 1.*—Change to read as follows:

1. This method of test covers a method for determining the potential reactivity of an aggregate with alkalies in portland-cement concrete as indicated by the amount of reaction during 24 hr at 80 C between 1 *N* sodium hydroxide and aggregate that has been crushed and sieved to pass a No. 50 sieve and be retained on a No. 100 sieve.

*New Section.*—Add a new Section 15 as follows:

*15. Interpretation of Results.*—Data correlating the results of this method with performance of aggregates in structures, expansion of mortar bars containing high-alkali cement, or petrographic examination have been published (**1-5**)[a] and should be reviewed before results of the method are used as a basis for conclusions and recommendations concerning the properties and use of aggregates for concrete. For most aggregates, a potentially deleterious degree of alkali reactivity is indicated if the plotted data point falls to the right of the boundary line shown in Fig. 2. The result so derived conforms closely with that obtained by application of criteria defined in Note 2, Section 4(*a*) of the Specifications for Concrete Aggregates (ASTM Designation: C 33).

Results of the test are not correct for some aggregates, primarily because of extraneous reduction of alkalinity (spurious increase of $R_c$ and/or decrease in $S_c$) produced by reaction of the sodium hydroxide with carbonates of magnesium or ferrous iron or certain silicates of magnesium such as antigorite (serpentine). In the presence of soluble silica, calcium carbonate may also cause small anomalous increases in $R_c$ and may either increase or decrease the values of $S_c$. The spurious changes of $R_c$ and $S_c$ caused by calcium carbonate affect the apparent degree of reactivity but an erroneous indication of potential reactivity is produced only if the degree of alkali reactivity of the aggregate is marginal.

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[a] The boldface numbers in parentheses refer to the references appearing at the end of this method.

In order to evaluate these possible effects, it is recommended that performance of the method be correlated with petrographic examination in accordance with the Recommended Practice for Petrographic Examination of Aggregates for Concrete (ASTM Designation: C 295) or any other test method or field experience that is applicable.

### REFERENCES

(1) R. C. Mielenz and L. P. Witte, "Tests Used by the Bureau of Reclamation for Identifying Reactive Concrete Aggregates," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 1071 (1948) and discussion, p. 1105.

(2) R. C. Mielenz, K. T. Greene, and E. J. Benton, "Chemical Test for Reactivity of Aggregates with Cement Alkalies: Chemical Processes in Cement-Aggregate Reaction," *Proceedings*, Am. Concrete Inst., Vol. 44, p. 193 (1948).

(3) Wm. Lerch, "Studies of Some Methods of Avoiding Expansion and Pattern Cracking Associated with the Alkali-Aggregate Reaction," Symposium on Use of Pozzolanic Materials in Mortars and Concretes, *ASTM Special Technical Publication, No. 99*, p. 153 (1950).

(4) F. O. Slate, "Chemical Reactions of Indiana Aggregates in Disintegration of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 954 (1949).

(5) Wm. Lerch, "Chemical Reactions of Concrete Aggregates," *ASTM Special Publication No. 169*, p. 334 (1956).

*Figure 2.*—Add a new Fig. 2 showing the boundary line between the plotted data for satisfactory and unsatisfactory aggregates.

**Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 – 56 T):**[3]

**Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 – 56 T):**[3]

**Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 – 56 T):**[3]

**Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 – 56 T):**[3]

*Section 5 (b).*—Add the following as Note 4 in C 290, C 291, and C 310 and as Note 3 in C 292:

NOTE.—It is recommended that the fundamental longitudinal frequency be determined initially and as a check whenever question exists concerning the accuracy of determination of fundamental transverse frequency, and that the fundamental torsional frequency be determined initially and periodically as a check on the value of Poisson's ratio.

*Section 6 (a).*—In line 2, change "shall" to read "may."

### REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative status:

**Standard Method of Test for Scratch Hardness of Coarse Aggregate Particles (C 235 – 54):**[2] Revise as appended hereto.[1]

**Standard Method of Test for Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel (C 234 – 54):**[2] Revise as appended hereto.[1]

### ADOPTION OF TENTATIVES AS STANDARD WITH REVISIONS

The committee recommends that the Tentative Specifications for Concrete Aggregates (**C 33 – 56 T**)[3] be approved for reference to letter ballot of the

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[3] 1956 Supplement to Book of ASTM Standards, Part 3.

Society for adoption as standard with revisions as follows:

*Sections 4 (c) and 8 (b).*—In line 2, delete "frequent."

*Table I.*—Change "Oven-dry material coarser than No. 50 sieve, floating on a liquid having a specific gravity of 2.0" to read "Coal and lignite."

*Table III.*—Change "Oven-dry material floating on a liquid having a specific gravity of 2.0" to read, "Coal and lignite."

*Section 8 (a).*—Change "each determined on independent samples" to read "determined on a sample."

*Section 12 (j)*—Change to read as follows:

> (*j*) *Coal and Lignite.*—Method of Test for Lightweight Pieces in Aggregate (ASTM Designation: C 123), using a liquid of 2.0 specific gravity to remove the particles of coal and lignite.

## REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

### Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 – 55):[2]

*Section 4 (a).*—Change the fourth sentence to read, "The assembled mold and base plate shall be watertight; the base plate shall be at right angles to the axis of the cylinder; and shall be oiled with mineral oil before use."

*Section 6 (a).*—In the second sentence, delete "and shall be approximately at right angles to the axis of the specimens." Add a new third sentence to read, "The capped surface of the specimen in contact with the lower bearing block of the testing machine shall not depart from perpendicularity by more than 0.5 deg (approximately equivalent to $\frac{1}{8}$ in. in 12 in.) and the combined departure of the two capped surfaces from perpendicularity to the axis shall not exceed 3 deg."

*Section 7 (a).*—Change to read as follows:

> 7. (*a*) Storage condition during the first 24 hr have an important influence on strength development of concrete at all ages. During the first 24 hr all test specimens shall be stored under conditions which maintain the temperature immediately adjacent to the specimens in the range of 60 to 80 F (16 to 27 C) and prevent loss of moisture from the specimens. Storage temperatures may be regulated by means of ventilation or by evaporation of water from sand or burlap, or by using heating devices such as stoves, electric light bulbs, or thermostat-controlled heating cables.
>
> Specimens may be stored in tightly-constructed, firmly-braced wooden boxes, damp sand pits, temporary buildings at construction sites, under wet burlap in favorable weather, or by other suitable methods, provided the foregoing requirements limiting specimen temperature and moisture loss are met. The temperature record of the specimen may be established by means of relatively inexpensive maximum-minimum thermometers designed for field use.

*Section 7 (b).*—Change "65 to 75 F (18 to 24 C)" to read "70.4 to 76.4 F (21.3 to 24.7 C)."

### Standard Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 – 49):[2]

*Section 4 (a).*—In the fifth line of this paragraph, change "shall" to "should" following "compressive strength."

*Section 7 (e).*—Change "as prescribed in Section 4 of the Standard" to read "in accord with the applicable provisions of the."

*Section 7 (f).*—Delete the last two lines of the tabulation.

### Standard Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 – 49):[2]

*Section 2.*—Add as a new first sentence, "The testing machine shall conform to the requirements of Section 15 of Methods of Verification of Testing Machines (ASTM Designation: E 4)."

### Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 55):[2]

*Section 8 (a).*—Change the third sentence to read, "The assembled mold and base plate shall be watertight; the base plate shall be at right angles to the axis of the cylinder; and shall be oiled with mineral oil before use."

*Section 10 (a).*—In the second sentence, delete "and shall be approximately at right angles to the axis of the specimens." Add a new third sentence to read, "The capped surface of the specimen in contact with the lower bearing block of the testing machine shall not depart from perpendicularity by more than 0.5 deg (approximately equivalent to $\frac{1}{8}$ in. in 12 in.) and the combined departure of the two capped surfaces from perpendicularity to the axis shall not exceed 3 deg."

*Section 11.*—Change "65 to 75 F (18 to 24 C)" to read "70.4 to 76.4 F (21.3 to 24.7 C)."

### Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 42):[2]

*Section 3.*—Add at the end of the section the sentence, "Mechanical aids, if desired, may be employed to assist in achieving the specified saturated surface-dry condition."

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends for continuation without revision the following tentatives which have stood without revision for two or more years:

*Tentative Method of Test for:*

Volume Change of Concrete Products (**C 341 – 54 T**),

*Tentative Specifications for:*

Lightweight Aggregates for Structural Concrete (**C 330 – 53 T**), and
Lightweight Aggregates for Concrete Masonry Units (**C 331 – 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

## SUBCOMMITTEE ACTIVITIES

The titles, scopes, and membership of all subcommittees were reviewed. The titles of Subcommittees II-c and II-g were changed to "Elastic and Inelastic Properties of Concrete" and "Pore Structure of Aggregates and Concrete," respectively.

This report has been submitted to letter ballot of the committee, which consists of 148 active members commanding 113 votes plus 7 honorary members having voting privileges; 134 members returned their ballots, of whom 130 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. H. PRICE,
*Chairman.*

BRYANT MATHER,
*Secretary.*

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE C-11

## ON

# GYPSUM*

Committee C-11 on Gypsum held two meetings during the year: in Atlantic City, N. J., on June 21 and 22, 1956, and in Philadelphia, Pa., on February 6 and 7, 1957.

The committee consists of 37 members, of whom 34 are voting members; 13 are classified as producers, 9 as consumers, 12 as general interest, and 3 as consulting members.

### REVISIONS OF TENTATIVE

Committee C-11, jointly with Committee C-7 on Lime, recommends revisions as follows of the Tentative Specifications for Inorganic Aggregates for Use in Interior Plaster (**C 35 – 54 T**):[1]

*Section 3(a)*.—Change the third, fourth, and fifth values for perlite by volume max from "45, 85, and 95" to read "60, 95, and 98."

*Section 4(b)*.—Change "7½" to read "6."

The committee is recommending that it be granted sole jurisdiction for Specifications C 35.

### REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Specification for Gypsum Plasters (C 28 – 55),[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

*Section 1.*—Change "four" to read "five," and following the listing of Gypsum wood-fibered plaster, add "Gypsum bond plaster."

*Section 6.*—Change the heading "Compressing Strength" to read "Compressive Strength." In the first sentence delete the words "when dry."

*Section 9.*—Add the word "Ottawa" following "standard."

*Section 10.*—In the first line change "tested" to read "mixed," and in line 2 add the words "by weight" following "2 parts."

*Section 14.*—Delete the words "when dry."

*New Sections.*—Add the following new Sections 15, 16, and 17 covering gypsum bond plaster, renumbering the present Section 15 and subsequent sections accordingly:

#### GYPSUM BOND PLASTER

15. *Description.*—Gypsum bond plaster is calcined gypsum mixed at the mill with other ingredients to control working quality and setting time, and to adapt it for application as a bonding scratch-coat over monolithic concrete. Addition of water only is required on the job.

16. *Composition.*—Gypsum bond plaster shall contain not less than 93.0 per cent by weight of calcined gypsum (see Section 2) and not less than 2.0 per cent nor more than 5.0 per cent of hydrated lime.

17. *Time of Setting.*—Gypsum bond plaster shall set in not less than 2 nor more than 10 hr. It shall be tested for time of setting in ac-

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 3.

cordance with the Methods of Testing Gypsum and Gypsum Products (ASTM Designation: C 26)[2] except that a 300-g sample shall be added to sufficient water to produce a stiff yet workable mortar, and the mixture shall then be stirred for 1 min to an even, lump-free consistency.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 34 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. W. JOSEPHSON,
*Chairman.*

O. H. STOREY, JR.,
*Secretary.*

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-11 presented to the Society through the Administrative Committee on Standards tentative revisions of the Standard Specifications for Gypsum Plasters (C 28 – 55). The tentative revisions were accepted by the Standards Committee on August 26, 1957, and they appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

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[2] 1956 Supplement to Book of ASTM Standards, Part 3.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE C-12

## ON

## MORTARS FOR UNIT MASONRY*

Committee C-12 on Mortars for Unit Masonry held two meetings during the year: on June 20, 1956, in Atlantic City, N. J., and on February 7, 1957, in Philadelphia, Pa.

The committee consists of 43 members, of whom 14 are classified as producers, 7 as consumers, and 22 as general interest members.

The committee records with sorrow the death of F. O. Anderegg, Masonry Consultant of Sommerville, N. J., a member since 1932.

The scopes of Subcommittees II and III were revised to transfer the responsibilities for methods of test to Subcommittee II.

### NEW TENTATIVE

The committee recommends for publication as tentative the Specifications for Aggregates for Masonry Grout as appended hereto.[1]

### REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Mortar for Unit Masonry (**C 270 – 54**)[2] and continuation of the Specifications as tentative:

*Section 2(a).*—Add to this section, "Portland Blast-Furnace Slag Cement.—Type IS or ISA of the Tentative Specifications for Portland Blast-Furnace Slag Cement (ASTM Designation C 205)."

*Tables I and II.*—Delete the old mortar designations A-1, A-2, B, C, and D and change Footnote (*a*) to read, "Mortar type designations A-1, A-2, B, C, D are the former type designations in effect prior to 1954."

*New Section.*—Add a new Section 11 as follows, and renumber the present Section 11 as Section 12.

> 11. *Water Retention.*—Mortar of the materials and proportions used in the construction, mixed to an initial flow of 100 to 115, shall have a flow after suction of not less than 70 per cent.

### TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specifications for Aggregates for Masonry Mortar (**C 144 – 52 T**).

### EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Tentative Specifications for Mortar for Unit Masonry (**C 270 – 54 T**):[2]

*Section 1.*—Add to the end of the fourth paragraph, "Mortar shall be accepted only under one requirement."

*Section 7.*—In line 2, after the words "cubes of" add "laboratory prepared." Add to the end of the section, "Mortar mixed to a flow suitable for use in laying masonry units shall not be required to meet the strength given in Table I for the mortar specified."

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

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* Presented at the Sixtieth Annual Meeting fo the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

## SUBCOMMITTEE ACTIVITIES

*Subcommittee II on Research and Methods of Test* (P. L. Rogers, chairman) has improved a proposed test method for efflorescence of mortar so that the procedure is quantitative. Consideration is being given to recommending its publication as tentative.

*Subcommittee III on Specifications* (H. C. Plummer, chairman) is preparing an extensive revision of the Tentative Specification for Mortar for Reinforced Brick Masonry (C 161 – 44).

*Subcommittee IV on Aggregates* (D. L. Bloem, chairman) prepared the proposed Tentative Specifications for Aggregates for Masonry Grout.

This report has been submitted to letter ballot of the committee, which consists of 43 members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. COPELAND,
*Chairman.*

C. U. PIERSON, JR.,
*Secretary.*

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE C-13
## ON
## CONCRETE PIPE*

Committee C-13 on Concrete Pipe held one meeting during the year: in Chicago, Ill., on November 27 and 28, 1956.

The committee consists of 45 members, of whom 20 are classified as producers, 16 as consumers, and 9 as general interest members.

New subcommittees have been organized: Subcommittee VI on Concrete Drain Tile (P. W. Manson, chairman) and Subcommittee VII on Specifications for Non-Reinforced and Reinforced Perforated Concrete Pipe (H. Allen, chairman).

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Committee C-13 presented to the Society through the Administrative Committee on Standards the following recommendations:

*New Tentative Specification for:*

Reinforced-Concrete Culvert, Storm Drain, and Sewer Pipe (C 76 - 57 T),

*Withdrawal of Standard Specifications for:*

Reinforced-Concrete Sewer Pipe (C 75 - 56), and
Reinforced-Concrete Culvert Pipe (C 76 - 56).

These recommendations were accepted by the Administrative Committee on June 12, 1957.[1]

### REVISION OF TENTATIVES

The committee recommends revisions as follows of the Tentative Specifications for Reinforced Concrete Low-Head Pressure Pipe (C 361 - 55 T):[2]

*Section 3.*—Change the second and third sentences to read as follows:

> If agreed in advance, specimens not less than 4 ft long, manufactured in accordance with the same procedures as used on longer lengths, or specimens cut from standard pipe sections shall be acceptable for determining external load tests. Pipe sections successfully passing the testing procedure shall be accepted by the purchaser.

*Section 14.*—In the seventh sentence, change the word "layers" to read "separate cages."

*Section 16.*—Change to read as follows:

> 16. *Longitudinals.*—Each layer of circumferential reinforcement shall be assembled into a rigid cage supported by longitudinal bars which extend the full length of the pipe. The number of longitudinal bars provided in each cage, and the total area of longitudinal reinforcement for pipe sizes 12 in. through 36 in. in diameter shall be as shown in Table II. For pipe larger than 36 in. in diameter each cage shall have a minimum of longitudinal reinforcement equivalent to $\frac{1}{2}$ in. round steel rods at a maximum spacing of 30 in. center to center measured circumferentially. Where the pipe joint construction requires the use of a bell, the minimum number and area of bars shall be provided in the bell, either continuous with or spliced with the main longitudinal bars. The circumferential bars of each cage shall be spaced and supported by welding or tying each hoop to not less than the minimum number of bars above specified, and the latter shall be spaced at approximately equal distances around

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

the periphery of the cage. Spacer bars, chairs, or other methods shall be provided to maintain the reinforcement cage in proper position within the forms during the placement and consolidation of the concrete.

*Section 18 (a).*—Change the first sentence to read, "In joints formed entirely of concrete except for those surfaces within the gasket groove, all inside surfaces of the bell or outside surfaces of the spigot, or both, on which the rubber gasket may bear during the closure of the joint and at any degree of partial closure shall be parallel within 1 deg and have an angle of not more than two degrees with the longitudinal axis of the pipe."

*Section 21.*—Replace the third sentence with the following:

> The pipe shall be cured until 6 by 12-in. concrete test cylinders cured in the same manner as the pipe have attained a strength of at least 4000 psi. If type V cement or other slow-hardening cement is specified by the purchaser, the curing shall be continued until a cylinder strength of 3500 psi is attained.

*Section 30 (a).*—Add to the end of the first sentence, "except that in not more than 10 per cent of the pipe units of any one size to be installed in any one continuous reach of pipeline, up to two times the above listed permissible variations, will be accepted for not more than one-fourth the length of the pipe unit. Within this distance the net area of the pipe opening shall not be reduced by more than 4 per cent for pipe having internal diameters of 12 to 36 in., inclusive, or more than 3 per cent for pipe having internal diameters larger than 36 in., and the transitions to the restricted areas shall be gradual and smooth.

*Section 35.*—Replace the third sentence with the following:

> All pipe on which the ends have been spalled beyond the line of contact of the rubber gaskets and where the spalled area is greater in width, along the line of contact, than one-half the radius of the pipe or 12 in., whichever is the lesser, shall be rejected. Individual air holes in gasket-bearing areas of precast concrete pipe may be filled with a hand-placed, stiff, preshrunk 1:1 mortar of cement and fine sand with no other preparation than thorough washing with water. Such fillings shall be kept moist under wet burlap for at least 48 hr.

*Table II.*—In the column entitled "Minimum Number of Bars in Each Cage," change "3" to read "4" wherever it appears. Delete pipe diameters 39 in. through 96 in. and corresponding values in all columns.

## REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the Standard Specifications for Concrete Sewer Pipe (**C 14 – 56**)[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

*Section 3.*—In the Note to this section, change "Specifications C 14 – 55" to read "Section 20."

*Section 5.*—Add to the end of this Section, "or shall be Portland-Pozzolan Cement conforming to the requirements of the Specifications for Portland-Pozzolan Cement (ASTM Designation: C 340)."

*Section 8.*—Add to the end of the first sentence, "and Table II."

*Section 9.*—Change to read as follows:

> 9.(*a*) *Permeability and Hydrostatic Tests.*—When subject to the permeability test as specified in Section 19, the outer pipe surface of not less than 80 per cent of the pipe tested shall show no moist or damp spots at the end of the test period due to water passing through the walls of the pipe.
>
> (*b*) If subjected to the internal hydrostatic pressures described in Section 20, the pipe shall show no leakage. Moisture appearing on the surface of the pipe in the form of patches or beads adhering to the surface shall not be considered leakage.

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[3] 1956 Supplement to Book of ASTM Standards, Part 3.

*Section 18.*—Change the center heading preceding this section from “Permeability Test” to read “Permeability or Hydrostatic Test.” Change “tested for leakage under” to read “subjected to.”

*Section 19.*—In the third and fourth sentences change “leakage” to read “moist or damp spots on the outer surface of the pipe.”

TABLE I.—PHYSICAL AND DIMENSIONAL REQUIREMENTS OF STANDARD STRENGTH, BELL AND SPIGOT NON-REINFORCED CONCRETE SEWER PIPE.[a]

| Internal Diameter, in. | Minimum Thickness of Barrel, *T*, in. | Minimum Laying Length,[b] *L*, ft | Inside Diameter at Mouth of Socket,[c] $D_s$, in. | Depth of Socket, $L_s$, in. | Minimum Taper of Socket, $H:L_s$ | Minimum Thickness of Socket,[d] $T_s$ | Minimum Strength, lb per lin. ft | | Maximum Absorption, per cent |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | Three-Edge Bearing Method[e] | Sand Bearing Method[f] | |
| *1* | *2* | *3* | *4* | *5* | *6* | *7* | *8* | *9* | *10* |
| 4 | 9/16 | 2½ | 6 | 1½ | 1:20 | 3T/4<br>all sizes | 1000 | 1500 | 8 |
| 6 | ⅝ | 2½ | 8¼ | 2 | 1:20 | | 1100 | 1650 | 8 |
| 8 | ¾ | 2½ | 10¾ | 2¼ | 1:20 | | 1300 | 1950 | 8 |
| 10 | ⅞ | 3 | 13 | 2½ | 1:20 | | 1400 | 2100 | 8 |
| 12 | 1 | 3 | 15¼ | 2½ | 1:20 | | 1500 | 2250 | 8 |
| 15 | 1¼ | 3 | 18¾ | 2½ | 1:20 | | 1750 | 2620 | 8 |
| 18 | 1½ | 3 | 22¼ | 2¾ | 1:20 | | 2000 | 3000 | 8 |
| 21 | 1¾ | 3 | 25¾ | 2¾ | 1:20 | | 2200 | 3300 | 8 |
| 24 | 2⅛ | 3 | 29½ | 3 | 1:20 | | 2400 | 3600 | 8 |

[a] See Fig. 5 also.

[b] Shorter lengths may be used for closures and specials.

[c] When pipe are furnished having an increase in thickness over that given in column 2, then the diameter *at the inside* of the socket shall be increased by an amount equal to twice the increase of the barrel.

[d] This measurement shall be taken ¼ in. from the outer end of the socket.

[e] See Section 15.

[f] See Section 16.

*New Section.*—Add a new Section 20 to read as follows, renumbering the subsequent sections accordingly:

20.(*a*) *Hydrostatic Test.*— The ends of the pipe shall be tightly closed by wooden or metallic bulkheads or covers faced with rubber or leather so that no leakage shall occur through the covers at the test pressure. One cover shall be provided with a ¾-in. nipple passing through the cover and held securely in place and made water-tight by means of locknuts and washers or gaskets. The outer end of the nipple shall be connected with a pump discharge or water service line.

(*b*) Water pressures, as measured by a standardized gage attached to the delivery pipe close to the specimen, shall be internally applied to the specimen as follows:

5 psi for 5 min
10 psi for 10 min
15 psi for 15 min

*Section 21.*—Renumber as Section 22 and change to read as follows:

22. Pipe shall be furnished of the sizes, internal diameters, and dimensions prescribed in Tables I, II, and III.

*Section 22.*—Renumber as Section 23 and change to read as follows:

23. The permissible variations from the dimensions prescribed in Tables I and II shall not exceed those stated in Table III. This is not to be construed, however, that heavier wall thickness pipe cannot be furnished at the option of the manufacturer. Pipe intended to be straight shall not have variation in alignment of more than ⅛-in. per ft of length.

*Section 25.*—Renumber as Section 26. In Paragraph (*a*), the first sentence, change “of the pipe” to read “ of all pipe.” In the second sentence, change “cementing” to read “jointing.”

In Paragraph (*b*), change "Unless otherwise specified," to read "Extra-strength."

*New Section.*—Add a new Section 28 to read as follows:

28. *Repairs.*—Pipe may be repaired, if necessary, because of occasional imperfections in manufacture or accidental injury during handling and will be acceptable if, in the opinion of the purchaser, the repairs are sound and properly finished and cured and the repaired pipe conforms to the requirements of these specifications.

TABLE II.— PHYSICAL AND DIMENSIONAL REQUIREMENTS OF EXTRA STRENGTH NON-REINFORCED CONCRETE SEWER PIPE.[a]

| Internal Diameter, in. | Minimum Thickness of Barrel,[b] in. | Minimum Laying Length,[c] ft | Minimum Strength. lb per lin. ft | | Maximum Absorption, per cent |
|---|---|---|---|---|---|
| | | | Three-edge Bearing Method[d] | Sand Bearing Method[e] | |
| *1* | *2* | *3* | *4* | *5* | *6* |
| 4 | 3/4 | 2 1/2 | 2000 | 3000 | 8 |
| 6 | 3/4 | 2 1/2 | 2000 | 3000 | 8 |
| 8 | 7/8 | 2 1/2 | 2000 | 3000 | 8 |
| 10 | 1 | 3 | 200C | 3000 | 8 |
| 12 | 1 3/8 | 3 | 2250 | 3375 | 8 |
| 15 | 1 5/8 | 3 | 2750 | 4175 | 8 |
| 18 | 2 | 3 | 3300 | 4950 | 8 |
| 21 | 2 1/2 | 3 | 3850 | 5775 | 8 |
| 24 | 3 | 3 | 4000 | 6000 | 8 |

[a] See Section 26 (*a*) and (*b*).

[b] Where tongue and groove pipe is furnished, it shall have a minimum shell thickness of 1 3/4 in. except that the above minimum thicknesses apply if the groove end is flared to give a groove thickness equal to 3/4 the thickness of the barrel of the pipe. This measurement shall be taken 1/4 in. from the outer end of the groove.

[c] Shorter lengths may be used for closures and specials.

[d] See Section 15.

[e] See Section 16.

*Section 27.*—Renumber as Section 29. In the first sentence before the word "initials" add "class of pipe."

*Tables I, II, and III.*—Change to read as shown in the accompanying Tables I, II, and III.

*Figure 5.*—In the caption, change "Table II" to read "Table I."

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specifications for Reinforced Concrete Low-Head Internal Pressure Sewer Pipe (**C 362 – 55 T**), and tentative revisions of the Standard Specifications for Concrete Pipe for Irrigation or Drainage (**C 118 – 55**).

TABLE III.—PERMISSIBLE VARIATIONS IN DIMENSIONS OF NON-REINFORCED CONCRETE SEWER PIPE.

| Nominal Size. Internal Diameter, in. | Limits of Permissible Variation in: | | | | |
|---|---|---|---|---|---|
| | Length, in. per ft[a] (–) | Length of Two Opposite Sides, in. | Internal Diameter, Pipe or Socket,[a] [b] in. (±) | Depth of Socket, in.[ab] (–) | Thickness of Barrel, in.[a] (–) |
| 4 | 1/4 | 1/4 | 1/8 | 1/8 | 1/16 |
| 6 | 1/4 | 1/4 | 3/16 | 1/4 | 1/16 |
| 8 | 1/4 | 5/16 | 1/4 | 1/4 | 1/16 |
| 10 | 1/4 | 3/8 | 1/4 | 1/4 | 1/16 |
| 12 | 1/4 | 3/8 | 1/4 | 1/4 | 1/16 |
| 15 | 1/4 | 7/16 | 1/4 | 1/4 | 3/32 |
| 18 | 1/4 | 1/2 | 1/4 | 1/4 | 3/32 |
| 21 | 1/4 | 9/16 | 5/16 | 1/4 | 1/8 |
| 24 | 3/8 | 9/16 | 5/16 | 1/4 | 1/8 |

[a] The minus sign – alone indicates that the plus variation is not limited; the plus and minus sign ± indicates variation in both excess and deficiency in dimension.

[b] Socket variation applies to standard strength bell and spigot pipe only.

## EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

### Standard Specifications for Concrete Sewer Pipe (C 14 – 56):[3]

*Figures 1 and 2.*—Change the details of the lower bearing wood strips to conform to the requirements of the test method.

*Figures 2 and 3.*—Change as recommended above for Figs. 1 and 2 of Specifications C 14.

**Standard Specifications for Concrete Pipe for Irrigation or Drainage (C 118 - 56):**[3]

*Figure 1.*—Change as recommended above for Fig. 1 of Specifications C 14.

**Tentative Specifications for Reinforced Concrete Low-Head Pressure Pipe (C 361 - 55 T):**[2]

*Figures 2 and 3.*—Change as recommended above for Figs. 1 and 2 of Specifications C 14.

**Tentative Specifications for Reinforced Concrete Low-Head Internal Pressure Sewer Pipe (C 362 - 55 T):**[2]

*Figures 2 and 3.*—Change as recommended above for Figs. 1 and 2 of Specifications C 14.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

## ACTIVITIES OF SUBCOMMITTEES

The subcommittees will pursue the recommendation of the main committee that those methods of test which are identical be removed from the specifications and published in a separate standard.

Subcommittees I, II, IV, and V will consider the matter of including a requirement for suitable types of rubber gaskets and the rubber composition for the gaskets in the specifications under their jurisdiction.

Recommendations of the Subcommittee on the Absorption Test were referred to Subcommittee II on Specifications for Reinforced-Concrete Sewer and Culvert Pipe. The Subcommittee on the Absorption Test will study comparable testing procedures in Iowa, Minnesota, Ohio. and Massachusetts during the year to correlate test results and determine their consistency.

Subcommittee VI on Concrete Drain Tile is preparing a tentative specification for concrete drain tile.

This report has been submitted to letter ballot of the committee, which consists of 45 members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
*Chairman.*

HOWARD F. PECKWORTH,
*Secretary.*

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE C-14
ON
# GLASS AND GLASS PRODUCTS*

Committee C-14 on Glass and Glass Products held two meetings during the year: on October 12, 1956, in Bedford Springs, Pa., and on May 8, 1957, in Dallas, Tex.

During the year there were 9 resignations from the committee, and 3 new members were added. The committee consists of 47 voting members, of whom 16 are classified as producers, 15 as consumers, and 16 as general interest members. There are 9 consulting members.

## ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision.

*Tentative Method of Test for:*
Average Linear Expansion of Glass (**C 337 – 54 T**),[1] and
Softening Point of Glass (**C 338 – 54 T**).[1]

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Annealing Point and Strain Point of Glass (**C 336 – 54 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 47 voting members; 26 members returned their ballots, of whom 25 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. G. GHERING,
*Chairman.*

F. V. TOOLEY,
*Secretary.*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE C-15
ON
# MANUFACTURED MASONRY UNITS*

Committee C-15 on Manufactured Masonry Units held two meetings during the year: on June 19, 1956, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society, and on February 8, 1957, in Philadelphia, Pa., during ASTM Committee Week.

The committee consists of 73 members of whom 35 are classified as producers, 18 as consumers, and 20 as general interest members.

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Tentative Revision of Standard Specifications for:*

Building Brick (C 62 – 50),
Hollow Load-Bearing Concrete Masonry Units (C 90 – 52),
Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 52),
Solid Load-Bearing Concrete Masonry Units (C 145 – 52), and

*Tentative Revision of Standard Methods of:*

Sampling and Testing Brick (C 67 – 50).

These recommendations were accepted by the Standards Committee on September 5, 1956, and the tentative revisions appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

## ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions[1] of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

*Standard Specifications for:*

Building Brick (**C 62 – 50**):[1] Revision of Section 2 and Explanatory Notes, issued September 15, 1955.
Facing Brick (**C 216 – 50**):[1] Revision of Section 4 and Explanatory Notes, issued September 15, 1955.
Structural Clay Load-Bearing Wall Tile (**C 34 – 55**):[1] Revision of Section 7, issued June, 1955.
Structural Clay Non-Load-Bearing Tile (**C 56 – 52**):[1] Revision of Section 7, issued June, 1955.
Structural Clay Floor Tile (**C 57 – 52**):[1] Revision of Section 7, issued June, 1955.

*Standard Methods of:*

Sampling and Testing Brick (**C 67 – 50**):[1] Revision of Section 15, issued September 15, 1955.

## REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision of the Standard Specifications for Facing Brick (**C 216 – 50**),[1] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

*Section 3.*—Reverse the order of the second and third paragraphs and change to read as follows:

*Type FBS.*—Brick for general use in exposed exterior and interior masonry walls and partitions where wider color ranges and greater variation in sizes are permitted than are specified for Type FBX.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 3.

*Type FBX*.—Brick for general use in exposed exterior and interior masonry walls and partitions where a high degree of mechanical perfection, narrow color range, and minimum permissible variation in size are required.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 73 members; 55 members have returned their ballots, of whom 53 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. W. WHITTEMORE,
*Chairman*.

M. H. ALLEN,
*Secretary*.

## EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

*New Tentative Specification for:*
Industrial Floor Brick (C 410 – 57 T)

*Revision of Tentative Specification for:*
Ceramic Glazed Structural Clay Facing Tile (C 126 – 55 T)

*Tentative Revision of Standard:*
Methods of Sampling and Testing Structural Clay Tile (C 112 – 52)
Specifications for Structural Clay Facing Tile (C 212 – 54)
Specifications for Facing Brick (C 216 – 50)

These recommendations were accepted by the Standards Committee on November 1, 1957, and the new and revised tentatives and tentative revisions of standards appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE C-16
## ON
# THERMAL INSULATING MATERIALS*

Committee C-16 on Thermal Insulating Materials held two meetings during the year: at Williamsburg, Va., on October 29 to 31, 1956, and at Philadelphia, Pa., on February 6 to 8, 1957, during ASTM Committee Week.

The committee sponsored a Symposium on Thermal Conductivity Measurements and Applications of Thermal Insulations during Committee Week. This Symposium will be printed as a separate publication *ASTM STP No. 216*.

At the last meeting, Ray Thomas, one of the original members of the committee and a past chairman, was elected to honorary membership.

A research project to determine the effects of moisture on the thermal conductivity of materials, sponsored by the committee at the Pennsylvania State University under the direction of Frank A. Joy, was recently completed and a final report on the project has been prepared. A summary of this report comprised the paper by Mr. Joy presented as part of the Symposium referred to above.

The committee consists of 93 voting members, 26 non-voting members, 4 honorary members, and 2 associate members. The voting membership is classified as follows: 47 producers, 27 consumers, and 19 general interest.

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations:

*New Tentative Specification for:*

Mineral Wool Block or Board Insulation for Elevated Temperature (**C 392 – 57 T**), and

*New Tentative Method of:*

Sampling Preformed Thermal Insulation (**C 390 – 57 T**).

These recommendations were accepted by the administrative committee on January 31, 1957, and March 6, 1957, respectively, and the new tentatives will appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

### NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Consistency of Wet-Mixed Thermal Insulating Cements as appended hereto.[1]

### REVISION OF TENTATIVE

The committee recommends revisions of the following tentatives as indicated and their continuation as tentative:

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

### Tentative Specification for Structural Insulating Board Made from Vegetable Fibers (C 208 – 55 T):[2]

*Tables I, II, and III.*—Change to read as shown in the accompanying Tables I, II, and III.[3]

### Tentative Methods of Testing Structural Insulating Board Made from Vegetable Fibers (C 209 – 55 T):[2]

*Section 35(b).*—Add the following note to this section:

NOTE.—If for some reason absolute ethyl alcohol is not available, Solox Anhydrous made by the U. S. Industrial Chemicals Co. has been found to give equivalent results and can be used.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee S-I on Block and Pipe Insulation* (P. Losse, chairman) has initiated work on the relation of these insulations to stress corrosion of austenitic stainless steel and the development of test methods to measure the handleability of block insulation. It is studying the development of product specifications for amosite asbestos fiber insulation and foamed plastic block and pipe insulation.

*Subcommittee S-II on Structural Insulating Board* (Wm. J. Marshall, chairman), in cooperation with industry groups, is working on specifications and test methods for structural insulating board, formboard and insulating roof deck.

*Subcommittee S-III on Insulating Cements* (E. J. Davis, chairman) is planning work on a specification for hydraulic-setting mineral-wool cement and methods of measuring consistency and corrosive tendencies of wet insulating cements.

*Subcommittee S-IV on Blanket Insulation* (J. M. Barnhart, chairman) is preparing a specification for batt or blanket type thermal insulation for buildings. Task groups have been appointed to consider preparation of a method of test for ruggedness of building insulation and definitions for felt, bulk and reflective type blankets.

*Subcommittee S-V on Loose-Fill Insulation* (L. A. Barron, chairman) broadened its scope to encompass foamed-in-place insulation and poured-in-place underground insulation. It is progressing on density determination methods for organic and inorganic loose-fill insulations.

*Subcommittee T-VI on Thermal Conductivity* (F. M. Gavan, chairman) has discontinued work on the D'Eustachio probe for determining thermal conductivity and rapid methods such as described recently by Land and Somers.

The present work consists of surveying commercial sources of thermal conductivity equipment, a revision of Method C 177 for Thermal Conductivity of Materials by Means of the Guarded Hot Plate, final approval of the very low temperature (Wilkes) method of determining thermal conductivity, methods of determining the thermal conductivity of mastics, and correlation of the definitions in the several methods of measuring thermal conductivity.

The subcommittee is preparing to work on the thermal conductivity of reflective insulations and methods of determining thermal conductivities at high temperatures.

*Subcommittee T-VII on Special Thermal Properties* (W. R. Seipt, chairman) is preparing methods of test for mechanical stability by abrasion of preformed thermal insulation, and tests for hot-surface performance, emissivity, fire resistance, and maximum use temperature limit.

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[2] 1955 Book of ASTM Standards, Part 3.

[3] These revised tables will be found in the revised specifications appearing in the 1957 Supplement to Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee S-VIII on Dimensional Standards* (H. P. Hoopes, chairman) is preparing recommended practices for field fabrication on thermal insulation fitting covers.

*Subcommittee T-IX on Vapor Transmission* (F. A. Joy, chairman) conducted a round-robin test on Methods of Test for Measuring Water Vapor Transmission of Materials in Sheet Form (E 96 – 53 T) and reported to Committee E-1 on Methods of Testing the discordant results traced to sealing methods. A proposed method of test for water vapor transmission was published in the ASTM BULLETIN. The subcommittee plans to develop a test method for vapor barriers used with insulation under slab floors on the ground.

*Subcommittee T-X on Physical Properties (Other than Permeance) of Coatings Accessory to Thermal Insulation* (T. A. Johnson, chairman) developed terms describing heat stability properties of coatings for thermal insulation and a method for preparing test specimens of mastic coatings.

*Subcommittee S-XI on Reflective Insulations* (G. E. Gronemeyer, chairman) is preparing definitions of basic terms for inclusion in the Standard Definitions of Terms Relating to Thermal Insulating Materials (C 168 – 56). Requests for methods of testing have been made to appropriate subcommittees, and the first material specification is being considered.

This report has been submitted to letter ballot of the committee, which consists of 93 voting members; 82 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

W. L. GANTZ,
*Chairman.*

J. M. HIGH,
*Secretary.*

# REPORT OF COMMITTEE C-18
# ON
# NATURAL BUILDING STONES*

Committee C-18 on Natural Building Stones held one meeting during the year: on April 16, 1957, at Washington, D. C. A meeting of the Advisory Subcommittee was held at Washington, D. C., on February 20, 1957. Subcommittee IV on Specifications met concurrently with the general meeting of the committee.

Committee C-18 consists of 22 members and 2 consulting members, of whom 6 are classified as producers, 10 as consumers, and 8 as general interest members.

## PROPOSED SPECIFICATION TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Proposed Specification for Structural Granite as appended hereto.[1]

## NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Roofing Slate as appended hereto.[2]

## TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Combined Effect of Temperature Cycles and Weak-Salt Solutions on Natural Building Stone (**C 218 – 48 T**), which has stood for more than two years without revision, be continued as a tentative.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

## SUBCOMMITTEE ACTIVITIES

*Subcommittee IV on Specifications* (F. S. Eaton, chairman).—The several work groups which had been set up to develop specifications for granite, limestone, marble, sandstone, and slate either met or have corresponded during the year. The granite work group has prepared a proposed specification for structural granite which is referred to above for publication as information only; the marble work group is preparing a specification; the slate work group has completed a proposed tentative specification for roofing slate which is referred to earlier in this report.

This report has been submitted to letter ballot of the committee, which consists of 21 members; 19 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

L. W. CURRIER,
*Chairman.*

F. S. EATON,
*Secretary.*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] See p. 278.

[2] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

[3] 1955 Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

## APPENDIX

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## PROPOSED SPECIFICATION FOR STRUCTURAL GRANITE[1, 2]

This is a proposed specification and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This specification covers the selection, sampling, and testing of granite for specific construction uses.

### General Characteristics

2. (*a*) The granite shall be free of starts, cracks, or similar imperfections that would impair its structural integrity.

(*b*) The designer shall specify the color desired and the permissible natural variations in color and texture in careful detail or by naming granites having the required characteristics. Granite containing minerals that cause objectionable stain upon exposure should be excluded.

### Physical Requirements

3. (*a*) *Test Requirements.*—The granite shall conform to the physical requirements prescribed in Table I. The designer shall select the grade and life expectancy of granite from Table I that will satisfy his requirement.

(*b*) *Service Requirements.*—Granite that has performed satisfactorily under environmental conditions similar to those of the intended use for a period of 50 yr or more may be exempted from the requirements prescribed in Paragraph (*a*) at the option of the designer.

### Sampling and Testing

4. (*a*) *Sampling.*—The granite shall be sampled in accordance with Section 2 of the Method of Test for Compressive Strength of Natural Building Stone (ASTM Designation: C 170).[3]

(*b*) *Compressive Strength.*—Determine the compressive strength of the granite in accordance with Sections 3, 4, 5(*a*), 6, 7, and 8 of Method C 170.

(*c*) *Abrasion.*—Determine the resistance to abrasion in accordance with Sections 2, 3, 4—Grading A, 5, and 6 of the Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (ASTM Designation: C 131).[3]

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[1] This proposed specification is under the jurisdiction of the ASTM Committee C-18 on Natural Building Stones.

[2] Published as information, June, 1957.

[3] 1955 Book of ASTM Standards, Part 3.

TABLE I.—PHYSICAL REQUIREMENTS.

| Specific Use | Life Expectancy | | | |
|---|---|---|---|---|
| | Less than 50 yr | | More than 50 yr | |
| | Compressive Strength, min, psi | Percentage of Wear, max | Compressive Strength min, psi | Percentage of Wear, max |
| *Engineering Grade:* | | | | |
| Bridge piers, sea and river walls, dams, and related structures | 25 000 | 35 | 30 000 | 32 |
| Bridge superstructures, grade separations, and retaining walls | 25 000 | 35 | 30 000 | 32 |
| Flexural members[a] | 30 000 | 32 | 30 000 | 32 |
| Traffic controls, curbs, etc. | 25 000 | 35 | 30 000 | 32 |
| *Architectural Grade:* | | | | |
| Monumental buildings | 28 000 | 34 | 30 000 | 32 |
| Institutional buildings | 26 000 | 36 | 28 000 | 34 |
| Commercial buildings | 20 000 | 40 | 26 000 | 36 |
| Residential buildings | 16 000 | 45 | 20 000 | 40 |
| Landscaping, parks, etc. | 25 000 | 35 | 30 000 | 32 |

[a] Modulus of rupture should be not less than 2000 psi according to the Method of Test for Modulus of Rupture of Natural Building Stone (ASTM Designation: C 99).[3]

# REPORT OF COMMITTEE C-19

ON

# STRUCTURAL SANDWICH CONSTRUCTIONS*

Committee C-19 on Structural Sandwich Constructions held two meetings during the year: on September 19, 1956 in Los Angeles, Calif. in conjunction with the Second Pacific Area National Meeting, and on April 23 to 25, 1957 at Massachusetts Institute of Technology, Cambridge, Mass.

In connection with its September meeting, the committee sponsored a Symposium on Sandwich Construction at which seven papers were presented.

The committee consists of 78 members of whom 64 are voting members; 33 are classified as producers, 10 as consumers, and 21 as general interest members.

## NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

*Method of Test for:*

Flatwise Flexure Strength of Sandwich Construction, and

Shear Fatigue of Sandwich Core Materials.

## ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

*Tentative Methods of Test for:*

Delamination Strength of Honeycomb Type Core Material (**C 363 – 55 T**),[2]

Edgewise Compressive Strength of Flat Sandwich Construction (**C 364 – 55 T**),[2]

Flatwise Compressive Strength of Sandwich Cores (**C 365 – 55 T**),[2] and

Measurement of Thickness of Sandwich Cores (**C 366 – 55 T**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

## SUBCOMMITTEE ACTIVITIES

*Subcommittee I on Mechanical Properties of Basic Materials* (S. Yurenka, chairman) is reviewing the standard methods for density (C 271 – 53) and for water absorption (C 272 – 53) of core materials to determine the need for revision. It recommended the adoption as standard of the tentative methods for flatwise compressive strength (C 365 – 55 T), delamination strength (C 363 – 55 T) and for measurement of thickness (C 366 – 55 T) of core materials referred to earlier in this report, and it prepared a method for impact strength of core materials. A method of test for water migration of core materials is in preparation.

*Subcommittee II on Properties of Basic Sandwich Construction* (E. W. Kuenzi, chairman) prepared the new proposed tentative flatwise flexure strength test method referred to earlier in the report. The subcommittee has recommended the use of the climbing drum peel test method for adhesives as developed by Committee D-14 on Adhesives. Cosideration is being given to two other peel test methods as alternate and more rapid methods a flexure creep test method, an impact test method and a form of nondestructive test method. Other properties of sandwich construction that have been suggested for study are vibration characteristics, sound absorption and transmission, and thermal properties.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 4.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee III on Permanence, Durability, and Simulated Service* (J. H. Gibbud, chairman).—Sandwich panels exposed for two years at State College, Pa., and at Kure Beach, N. C. were removed for test during April of this year. Similar panels were tested last year after one year exposure and next year the last panels in this program will be tested after three years exposure. A second exposure program designated as "Beta" was started in April, 1957, and will have panels tested after one, two, and three years exposure at the same two sites.

A tentative procedure for laboratory aging is in preparation.

*Subcommittee IV on Nomenclature and Definitions* (R. A. Biggs, chairman) has appointed a task group (F. J. McGarry, chairman) to prepare a list of standard symbols.

This report has been submitted to letter ballot of the committee, which consists of 65 voting members; 45 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

GEORGE GERARD,
*Chairman.*

W. L. EMERSON,
*Secretary.*

# REPORT OF COMMITTEE C-20
ON
# ACOUSTICAL MATERIALS*

Committee C-20 on Acoustical Materials held two meetings during the year: at the University Club of Chicago, Ill., on October 9, 1956, and at the American Standards Association Headquarters in New York City on May 2, 1957.

The committee consists of 75 members, of whom 24 are classified as producers, 13 as consumers, 29 as general interest members, and 9 as consulting members.

## ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Methods of Test for Strength Properties of Prefabricated Architectural Acoustical Materials (**C 367 - 55 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard.

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Sound Absorption* (H. J. Sabine, chairman).—The first draft of a test method for the measurement of sound absorption in reverberation rooms was circulated for comments and suggestions. Other projects include a box method for absorption testing, now in the first draft stage, and a study of the "horn coupler" modification of the impedance tube method by which specimens larger than the tube diameter may be tested.

*Subcommittee II on Flame Resistance* (Wallace Waterfall, chairman).—The fourth draft of the test method for flame resistance classification of acoustical materials based on Federal Specification SS-A-118b is being studied in conjunction with round robin tests of a task group. Studies of the radiant panel method and the large and small tunnel methods are continuing.

*Subcommittee III on Maintenance* (W. C. Clark, chairman).—The investigation of suitable test methods to evaluate the repaintability and cleansability of acoustical materials is continuing. A horn coupler for the Bureau of Standards impedance tube has been constructed to permit acoustical tests on full size painted specimens 1 ft square.

*Subcommittee IV on Application* (L. F. Yerges, chairman).—A draft specification and test method for acoustical adhesives will be circulated to committee letter ballot upon approval by Committee D-14 on Adhesives, joint sponsor. Two task groups were appointed to explore the problems of plastic acoustical materials (such as sprayed plaster) and to survey methods of mechanical attachment of acoustical tile.

*Subcommittee V on Basic Physical Properties* (W. A. Jack, chairman).—Studies of a method of light reflectance measurement based on the Baumgartner sphere have led to a round robin series of measurements of five light reflection plaques furnished by the Bureau of Standards. A program sponsored by the Acoustical Materials Assn. on the transmission of sound through suspended acoustical ceilings is being followed by the subcommittee for the possible development of an ASTM method.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 4.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 66 voting members; 32 members returned their ballots, of whom 31 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. A. LEEDY,
*Chairman.*

J. W. GARRISON,
*Secretary.*

# REPORT OF COMMITTEE C-21

## ON

# CERAMIC WHITEWARES AND RELATED PRODUCTS*

Committee C-21 on Ceramic Whitewares and Related Products held two meetings during the year: in Bedford Springs, Pa., on September 27, 1956, and in Dallas, Tex., on May 6, 1957.

The committee consists of 76 voting members, of whom 44 are classified as producers, 8 as consumers, and 24 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chariman, M. D. Burdick.
Vice-Chairman, G. W. Phelps.
Secretary, R. W. Cline.

## NEW TENTATIVES

The committee recommends for publication as tentative the following two methods as appended hereto:[1]

*Methods of Test for:*

Compressive (Crushing) Strength of Fired Whiteware Materials, and
Thermal Conductivity of Ceramic Whitewares.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Executive Subcommittee* (M. D. Burdick, chairman) completed the study of Reorganization plans designed to give increased status to groups working on product specifications.

*Subcommittee I on Nomenclature* (A. S. Watts, chairman) is considering 21 new definitions.

*Subcommittee II on Tests and Specifications* (V. E. Campbell, chairman) is considering a test for crazing resistance by the autoclave method developed by Subsection C-1.

It prepared the proposed tentative methods referred to earlier in the report. The Method for Compressive Strength was sponsored by the Engineering Ceramics Section, Mineral Products Division, National Bureau of Standards. The method for Thermal Conductivity was sponsored by the U. S. Signal Corps, Squier Laboratories, Ft. Monmouth, N. J., and the U. S. Air Force, Wright Air Development Center, Dayton, Ohio.

*Subsection A1 on Clays* (G. W. Phelps, chairman) is continuing the development of procedures for particle size determination, dry modulus of rupture determination for ceramic bond clays, and hydrogen-ion concentration measurements.

*Subsection A4 on Cermets* (T. S. Shevlin, chairman) has been concerned with establishing the scope of its future activity with relation to allied interests of other ASTM committees.

*Subsection A5 on Ferrites* (G. Economos, chairman) has completed a survey of the fields of interest to the ferrite manufacturer and consumer. The following seven categories were established: Low-frequency materials, T.V. materials, microwave application, transducer application, square loop materials, permanent magnets, and magnetic powders.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The definition for ferrites as nonmetallic magnetic materials was approved by a majority of the members present at the first meeting.

*Subsection C1* (W. C. Mohr, chairman) completed a tentative method of test for crazing resistance by autoclave treatment of fired whitewares.

*Subcommittee III on Research* (H. Z. Schofield, chairman) is continuing work on a method of measuring subsieve particle size. Projects being considered for investigation include resistance of overglaze colors to detergents, survey of methods for determining modulus of rupture, and methods for determination of degree of vitrification.

A Task Group on Nuclear Applications, J. M. Warde, chairman, will keep abreast of developments in this field.

This report has been submitted to letter ballot of the committee, which consists of 76 voting members; 57 members returned their ballots, of whom 55 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

M. D. BURDICK,
*Chairman.*

R. W. CLINE,
*Secretary.*

# REPORT OF COMMITTEE C-22

## ON

# PORCELAIN ENAMEL*

Committee C-22 on Porcelain Enamel held two meetings during the year: on October 2 and 3, 1956, in Columbus, Ohio, as guests of the Battelle Memorial Institute, and on February 4 and 5, 1957, in Philadelphia, Pa., during ASTM Committee Week.

The committee consists of 46 members, of whom 39 are voting members, with 22 classified as producers, 5 as consumers, and 12 as general interest members.

### NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Torsion Resistance of Laboratory Specimens of Porcelain-Enameled Iron and Steel as appended hereto.[1]

### REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[1] of the Tentative Definitions of Terms Relating to Porcelain Enamel (**C 286 – 56 T**).[2]

### ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Reflectivity and Coefficient of Scatter of White Porcelain Enamels (**C 347 – 54 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without change.

### TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following be continued as tentative without revision:

*Tentative Methods of Test for:*

Impact Resistance of Porcelain-Enameled Utensils (**C 284 – 51 T**), and

Warpage of Porcelain-Enameled Flatware (**C 314 – 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Research* (J. J. Canfield, chairman) has completed an investigation of possible test methods for determining the reboil characteristics of enameling iron and sheet steels and has reported results to Subcommittee IV.

*Subcommittee II on Nomenclature* (E. E. Howe, chairman) prepared the revision of the Tentative Definitions of Terms Relating to Porcelain Enamel (**C 286 – 56 T**) referred to earlier in the report. This work on definitions is approximately 75 per cent completed.

*Subcommittee III on Education* (L. S. O'Bannon, chairman) has continued its contacts with the technical press concerning the work of Committee C-22 and is preparing a brochure for distribution to some 300 companies interested in the porcelain enamel industry.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new and revised tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1956 Supplement to Book of ASTM Standards, Part 3.

[3] 1955 Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee IV on Materials* (H. S. Saunders, chairman) prepared the proposed Tentative Method of Test for Torsion Resistance of Laboratory Specimens of Porcelain-Enameled Iron and Steel referred to earlier in the report, and recommended the adoption of Tentative Method C 347.

*Subcommittee V on Finished Products* (J. C. Richmond, chairman) has work well under way on methods for the measurement of the resistance of porcelain enamels to abrasion and the resistance of porcelain enamels on aluminum to spalling. It recommended the continuation without revision of Tentative Methods C 284 and C 314, referred to earlier in the report.

This report has been submitted to letter ballot of the committee, which consists of 39 voting members; 29 members returned their ballots, of whom 28 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
*Chairman.*

G. H. SPENCER-STRONG,
*Secretary.*

# REPORT OF COMMITTEE D-1

ON

# PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Materials held two meetings during the year: on June 18 to 20, 1956, in Atlantic City, N. J., in connection with the Annual Meeting of the Society, and in Washington, D. C., on February 20 to 22, 1957.

At the June, 1956, meeting a Symposium on Floor Finishes was held. N. B. Garlock served as chairman and a discussion panel was made up of A. T. Bohnert, C. C. Hartman, and R. J. Phair. The papers and discussions covered the special requirements, formulation, application, testing, and maintenance of oleoresinous, synthetic resin, and shellac floor finishes.

At the February meeting, L. E. Rosenthal, General Motors Corp., presented an illustrated talk on "The Preparation of Technical Data for Presentation."

Committee D-1 participated in the Society's Second Pacific Area National Meeting held in Los Angeles, Calif., during the week of September 16, 1956, by sponsoring a Technical Session of eight papers which reported on the recent and current activities of a number of the subcommittees. The program served to introduce the committee's work and interests to the heavy concentration of paint manufacturers in southern California.

A new Subcommittee on Statistical Application was organized which is responsible for recommending statistical techniques for sampling materials, designing experiments, presenting data, and interpreting results and, on request, to review the adequacy of statistical procedures specified in existing or proposed methods of test.

A total of 61 standards have been approved jointly by the Federation of Paint and Varnish Production Clubs and the ASTM, cooperating through the Joint Committee composed of representatives appointed by the two parent organizations. An additional group of ASTM standards and tentatives is under active consideration by the Federation. Under the new operating procedure of the Federation, C. Homer Flynn, Executive Secretary and Editor of the *Official Digest*, has arranged for publication in the *Official Digest* of those ASTM standards which are under Federation consideration as joint standards.

The American Standards Assn. has approved as American Standard the following ASTM specifications and methods of analysis in their revised forms, which are under the jurisdiction of Committee D-1:

Specifications for Titanium Dioxide Pigments (ASTM D 476 - 48; ASA K45.1—1956), and
Methods of Chemical Analysis of White Pigments (ASTM D 34 - 56 T; ASA K15.1—1956).

The Subcommittee on Intercommittee Relations, composed of Committee D-1 representatives on other ASTM technical committees, is continuing to maintain close contact with those committees whose activities are of interest to Committee D-1.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16-21, 1957.

The committee approved an amendment to the Regulations Governing Committee D-1 to make provision for filling vacancies in the membership of the Advisory Committee.

At the February meeting, the committee voted unanimously to elect M. B. Chittick to Honorary Membership in Committee D-1.

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Tentative Specifications for:*

Refined Soybean Oil (D 1462 - 57 T).

*Tentative Method of:*

Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes and Related Materials (D 1466 - 57 T).

*Revision of Tentative:*

Recommended Practice for Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer and Related Products (D 822 - 46 T),

Method of Test for Permanganate Time of Lacquer Solvents and Diluents (D 1363 - 55 T), and

Method of Test for Effect of Household Chemicals on Clear and Pigmented Organic Finishes (D 1308 - 54 T).

*Tentative Revision of:*

Methods of Testing Varnishes (D 154 - 56).

*Revision and Reversion to Tentative of:*

Methods of Chemical Analysis of Dry Mercuric Oxide (D 284 - 33).

## NEW TENTATIVES

The committee recommends for publication as tentative the following nine methods of test as appended hereto:[1]

*Tentative Methods of Test for:*

Fatty Acids Used in Protective Coatings,

Volatile Matter in Tricresylphosphate,

Total Rosin Acids Content of Coating Vehicles,

Two-Parameter 60-deg Specular Gloss,

Color-Difference Using the General Electric Spectrophotometer,

Oil Absorption of Pigments by Gardner-Coleman Procedure,

Knoop Indentation Hardness of Paint, Varnish, and Lacquer Coatings,

Density of Paint, Varnish, Lacquer, and Related Products, and

Heptane Miscibility of Lacquer Solvents.

## REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following three methods and their continuation as tentative:

**Tentative Method of Test for Alcohol in Methyl Isobutyl Ketone (D 1362 - 55 T):**[2] Revise as appended hereto.[1]

**Tentative Method of Test for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood (D 1211 - 52 T):**[2] Revise as appended hereto.[1]

**Tentative Method of Test for Tinting Strength of White Pigments (D 332 - 55 T):**[2] Revise as follows:

*Section 2.*—Add the following as Paragraph (*e*), to cover the Hoover Muller, and reletter the present paragraphs accordingly:

> (*e*) *Hoover Muller.*—A Hoover Muller fitted with glass plates. The two glass plates shall be free of blowholes and other imperfections on the surface. Keep plates sharp by removing from machine and lightly grinding on a flat glass plate as in Paragraph (*c*) with No. 303 optical emery, or equivalent, and turpentine. When sharpening plates, rotate them at the end of each mull as per instructions on the use of the Glass Muller and Rubbing Slab.

*Section 5.*—Designate the present procedure as "Procedure A, Glass Muller."

*New Procedure.*—Add the following new "Procedure B, Hoover Muller," and renumber Section 6 as 7:

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[1] The new and revised tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 4.

[2] 1955 Book of ASTM Standards, Part 4.

6. *Procedure B, Hoover Muller:* (*a*) In order to minimize the effect of the difference in grinding of the area near the center of the plates as compared to the area near the periphery of the plates, draw two concentric circles under the base plate of the Hoover muller in such a way that they show clearly through the plate. These circles can be drawn either on a paper inserted under the plate, or drawn directly on the bottom of the plate. The inner circle should be $2\frac{1}{2}$ in. in diameter and the outer circle $4\frac{1}{2}$ in. in diameter.

(*b*) Carefully weigh the pigment as described in Procedure A, Section 5 (*a*), transferring the pigment to the base plate of the Hoover muller. Add the oil and work the pigment and oil into a paste with the spatula. Distribute the paste within the area between the two concentric circles on the plate, close the Muller and add a 50-lb weight (to make a total weight of 100 lb) pressing the plates together. Mull the paste with three series of mulls of 50 revolutions each with a pickup of the paste between each series spreading the paste as before between the two concentric circles before starting the next series.

(*c*) Continue the treatment of the paste of the sample and standard as described in Section 5, (*b*), (*c*), and (*d*).

## TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as indicated of the following standards:

### Standard Definitions of Terms Relating to Paint, Varnish, Lacquer and Related Products (D 16 – 52).[2]

Add the following new definitions of caulking compound and glazing compound:

*Caulking Compound.*—A soft plastic material, consisting of pigment and vehicles, used for sealing joints in buildings and other structures where normal structural movement may occur. It is available in several forms, such as gun, knife, and extruded preformed shapes.

*Glazing Compound.*—A dough-like material consisting of pigment and vehicle, used for sealing window glass in frames. It differs from putty in that it retains its plasticity for an extended period.

### Standard Methods of Testing Drying Oils (D 555 – 54):[2]

*Sections 10 to 13.*—Change the present test for Acid Number to read as follows:

#### ACID VALUE

10. *Definition: Acid Value* is the number of milligrams of potassium hydroxide required to neutralize the free acids in 1 g of sample.

11. *Apparatus: Erlenmeyer Flasks,* 250 to 300 ml.

12. *Reagent and Solvents:* (*a*) *Titrant.*—Approximately 0.1 *N* Potassium Hydroxide in Methyl Alcohol).—Use synthetic methyl alcohol conforming to ASTM Specifications D 1152. Standardize using potassium hydrogen phthalate (National Bureau of Standards Acid Potassium Phthalate No. 84) in methyl alcohol and 1 per cent phenolphthalein indicator.

(*b*) *Neutral Solvent Mixture.*—Mix equal parts of isopropyl alcohol conforming to ASTM Specifications D 770 and industrial toluol conforming to ASTM Specifications D 362. Neutralize using titrant as above and 1 per cent phenolphthalein indicator until pink color persists for 1 min.

(*c*) *Indicator* (1 per cent Phenolphthalein).—Dissolve 1 g of phenolphthalein in 100 ml of methyl alcohol conforming to ASTM Specifications D 1152.

*Procedure:*

13. (*a*) Transfer to an Erlenmeyer flask the weight of sample prescribed in the following table:

| Acid Value | Approximate Weight of Sample, g | Accuracy of Weighing, plus or minus, g |
|---|---|---|
| 0 to 5 | 20 | 0.05 |
| 5 to 15 | 10 | 0.05 |
| 15 to 30 | 5 | 0.05 |
| 30 to 100 | 2.5 | 0.001 |
| 100 and over | 1.0 | 0.001 |

(*b*) Add 100 ml of neutral solvent. Mix until dissolved.

(*c*) Add 1 ml of indicator and titrate until pink color persists for 1 min.

14. *Calculation:* Calculate as follows the acid value as the weight in milligrams of potassium hydroxide required to neutralize 1 g of sample:

$$\text{Acid value} = \frac{AB}{W}$$

where:

$A$ = milliliters of KOH solution required for titration,

$B$ = milligrams of KOH per milliliter of titrant, and

$W$ = weight of sample in grams.

## ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Effect of Household Chemicals on Clear and Pigmented Organic Finishes (**D 1308 - 54 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

## ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends for adoption as standard the Test for Acid Number of Bleached Lac and Bleached Lac Varnish described in Sections 6 to 13 of the Methods of Testing Shellac (**D 29 - 54 T**),[2] and incorporation of this procedure in the Standard Methods of Sampling and Analysis of Shellac (**D 29 - 40**) as Sections 39 to 46, revised as follows. The test for color of orange shellac in Sections 1 to 5 is to be continued as tentative.

*Section 7.*—In the third line, delete the words "the free acids in"; in the fourth line, delete the word "in."

*Footnote 4.*—In the third line, change "1950" to "1955."

*Section 9 (b).*—Change to read as follows:

> (*b*) *Standard Aqueous or Alcoholic Potassium Hydroxide or Sodium Hydroxide Solution (0.1 N):*
>
> (1) *Preparation of aqueous solution.*—Prepare a stock concentrated solution by dissolving KOH or NaOH in water in the proportion of 112 g of KOH or 85 g of NaOH in 200 ml of water. Allow the solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the carbonate precipitate into another clean bottle. Add clear $Ba(OH)_2$ to solution until no further precipitate forms. Again allow to settle until clear. Draw off 175 ml and dilute to 10 liters with water. Preserve in a stock bottle provided with a guard tube filled with soda lime.
>
> (2) *Preparation of alcoholic solution.*—Place 5 to 10 g of KOH in a 2 liter flask and add 1 to 1.5 liters of ethyl alcohol (95 per cent) or specially denatured alcohol conforming to Formula 1 or 3A of the U. S. Bureau of Internal Revenue. Boil on a water bath under a reflux condenser for 30 to 60 min. Distill and collect the alcohol. Dissolve 5.6 g of KOH or 4 g of NaOH in 1 liter of the distilled alcohol, keeping the temperature below 15.5 C while the alkali is being dissolved. This solution should remain clear.
>
> (3) *Standardization of solution.*—Standardize either solution by titrating against pure acid potassium phthalate using phenolphthalein indicator. The solution will be approximately 0.10 *N*. Determine its exact normality and make proper corrections in using it.

## REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following three standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

### Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 52):[2]

Change the definition of putty to read as follows:

> **Putty.**—A dough-like material consisting of pigment and vehicle, used for sealing glass in frames, and for filling imperfections in wood or metal surfaces. See Glazing Compound.

### Specifications for Orange Shellac and Other Lacs (D 237 - 55):[2]

*Table I.*—For "Matter insoluble in specified hot solvents, max. per cent," under Grade A, change "1.25" to "1.00," and under Grade C, change "1.75" to "1.50."

In the line "Iodine number, max.," under Grade D, change "24.5" to "15."

In the line "Rosin, per cent," delete "per cent" and under Grade D insert "none."

### Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 53):[2]

*Section 8.*—Replace with a reference to the Tentative Method of Test for

Heptane Miscibility of Lacquer Solvents.[1]

*Section 20.*—Replace with a reference to the Tentative Method of Test for Volatile Matter in Tricresyl Phosphate.[1]

### WITHDRAWAL OF TENTATIVES

The committee recommends the withdrawal of the following tentatives:

*Tentative Methods of Test for:*

Ester Value of Tricresyl Phosphate (**D 268 - 49 T**),[2] and
Purity of Acetone and Methyl Ethyl Ketone (**D 1154 - 54 T**).[2]

These two methods are no longer required by the Specifications for Methyl Ethyl Ketone (D 740 - 56), for Tricresyl Phosphate (D 363 - 56), or by the Tentative Specifications for Acetone (D 329 - 55 T).

### EDITORIAL REVISIONS

The committee recommends editorial changes as indicated in the following two standards and one tentative:

**Methods of Chemical Analysis of White Linseed Oil Paints (D 215 - 41):**[2]

*Section 30.*—Add a footnote to read as follows to the test procedure for matter soluble in water:

> The water-soluble content of composite pigmentation, as determined by this method, is frequently higher than the sum of the water-soluble matter in the individual pigments. Possibly this is due to reaction in water between the individual pigments.

**Method of Test for Color of Clear Liquids (Platinum-Cobalt Scale) (D 1209 - 54):**[2]

*Section 2(a).*—Change the specified dimension of the Nessler tubes from "300 ± 3 mm" to read "275 to 295 mm." This change conforms to manufacturer's specifications.

**Tentative Method of Test for Water in Lacquer Solvents and Diluents (Fischer Reagent Titration Method) (D 1364 - 55 T):**[2]

*Footnote 5.*—Change "Junior Titrimeter" to read "Fisher Titrimeter." The first-named apparatus is no longer manufactured.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee II on Drying Oils* (D. S. Bolley, chairman) prepared the proposed Tentative Specifications for Refined Soybean Oil, the proposed Tentative Method of Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnish and Related Materials, the proposed Tentative Methods of Testing Fatty Acids Used in Protective Coatings (for Acid Value, Color, Iodine Value, Saponification Value, Titer, and Unsaponifiable Matter). The subcommittee also prepared the proposed tentative revision of Sections 10 to 13 on Acid Value of Standard Methods of Testing Drying Oils (D 555 - 54). With the approval and cooperation of Committee D-17 on Naval Stores, the Working Group on Fatty Acids has expanded the scope of its work to include tall oil fatty acids; Committee D-17 has appointed a representative to work with the group. It is also drafting specifications for fatty acids. The subcommittee is continuing its development of a procedure for determining total unsaturation of conjugated oils. New groups have been organized to consider revision of the Specifications for Boiled Linseed Oil (D 260 - 55), for Raw Linseed Oil (D 234 - 55); and for Raw Tung Oil (D 12 - 55).

*Subcommittee III on Bituminous*

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Emulsions* (R. H. Cubberley, chairman) has determined that there is no demand for specifications for coal-tar emulsions for use as protective coatings for metals.

*Subcommittee IV on Traffic Paint* (W. W. Burr, chairman) is investigating the need for revision of the Method of Test for No-Pick-Up Time of Traffic Paint (D 711 - 55). It is continuing its road tests in cooperation with state highway departments and is drafting a method for the measurement of daylight reflectance of glass beads.

*Subcommittee V on Volatile Solvents for Organic Protective Coatings* (M. B. Chittick, chairman) is completing its work on a method to be proposed for testing for copper corrosion by mineral spirits. It has also undertaken revision of the Tentative Methods of Test for Kauri-Butanol Value of Hydrocarbon Solvents (D 1133 - 54 T), and for Aniline Point and Mixed Aniline Point of Hydrocarbon Solvents (D 1012 - 51).

The subcommittee will cooperate with Technical Subcommittee 66 of the New York Paint and Varnish Production Club in its work on the comparative evaporation rates of paint solvents.

*Subcommittee VI on Definitions* (G. G. Sward, chairman) prepared the proposed tentative definitions for caulking compound and glazing compound and the proposed revision of the definition for putty in the Definitions Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 52). The new terms and the revision were prepared in cooperation with Subcommittee XIX on Putty, Glazing, and Caulking Compounds.

*Subcommittee VII on Accelerated Tests for Protective Coatings* (H. D. Pharo, chairman) prepared the proposed revision of the Tentative Recommended Practice for Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products (D 822 - 46 T), to bring it in conformance with the Recommended Practice for Operation of Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Artificial Weathering Test (E 42 - 55 T). The subcommittee is completing its work on methods for the preparation of aluminum test panels and for the preparation of tin-plate test panels. It is actively working also on a method for water-fog testing, on the yellowing of interior architectural finishes, and on the staining of transportation finishes. With Subcommittee XXV on Cellulosic Coatings and Related Materials, it has been made jointly responsible for the Tentative Method of Test for Effect of Household Chemicals on Clear and Pigmented Organic Finishes (D 1308 - 54 T). The subcommittee has established new groups to work on cold-crack tests of automotive finishes on metal substrates and on a method of rating panels subjected to salt-spray test.

*Subcommittee VIII on Methods of Chemical Analysis of Paint Materials* (W. H. Madson, chairman) prepared the proposed revision and recommended reversion to tentative of the Method for Chemical Analysis of Dry Mercuric Oxide Pigment (D 284 - 33), and also recommended the addition of the explanatory footnote to the Methods for Chemical Analysis of White Linseed Oil Paints (D 215 - 41), referred to above. It is studying the method for determining per cent volatility of paints, the determination of water-soluble salt content by conductivity methods, and is working to improve and clarify the Tentative Methods for Chemical Analysis of White Titanium Pigments (D 1394 - 56 T).

*Subcommittee IX on Varnishes* (J. C. Weaver, chairman) prepared the proposed Tentative Methods of Test for Total Rosin Acids Content, for Gas Checking of Varnish Films, and for Draft Test of Varnish Films. The subcommittee also prepared the proposed revision of the "Tentative Revision" of the Standard Methods of Testing Varnishes (D 154 – 56) which involved changes in the specification of the numerical definition of three Gardner color standards. The subcommittee is preparing a method of test for skinning, an omnibus specification for driers, a revision of the bubble tube - timer method for determination of viscosity of transparent liquids, and on the analysis of and specifications for phthalic anhydride. In addition to the tentative gas checking and draft tests mentioned above, the subcommittee is also working on a qualitative test for rosin and on a test for ash content; these four methods are all based on methods in Federal Specification TT-P-141. A new group is investigating the possibility of developing permanent standards for the comparison of color of transparent dry films applied on milk glass or similar substrates.

A summary of the results of Collaborative Viscosity Tests Conducted by Group 18 appears as an Appendix to this report.[4]

*Subcommittee X on Optical Properties* (H. K. Hammond, III, chairman) prepared the proposed Tentative Methods of Test for Two-Parameter 60-deg Gloss, for Color Difference Using the General Electric Spectrophotometer, and for Color Difference Using the Color Eye. It also prepared the revision of the Tentative Method of Test for Tinting Strength of White Pigments (D 332 – 55 T), which makes provision for the use of the Hoover-Muller as an alternate procedure for pigments dispersion. The subcommittee is completing its work on new methods of specifying color by the Munsell system, for color difference using the Color Master Colorimeter, and for definitions for six appearance terms. The Tentative Method of Test for Specular Gloss (D 523 – 55 T) is under reexamination to determine if the specifications for source and receptor apertures are too stringent. It is continuing its work on hiding power and on illuminating and viewing conditions for visual color matching.

*Subcommittee XI on Resins* (C. F. Pickett, chairman) is bringing to completion its work on new methods for the determination of polyhydric alcohols, for a softening mold method of preparing a sample for the ball-and-ring method for softening point, for melamine content of nitrogen resins, and a revision of the Tentative Method of Test for Unsaponifiable Matter of Alkyd Resins and Resin Solutions (D 1397 – 56 T). When completed, the softening mold method mentioned above will be submitted to Committee E-1 on Methods of Testing with the recommendation that it be included in the Tentative Method of Test for Softening Point by Ring and Ball Apparatus (E 28 – 51 T). Other work is under way on the determination of phthalic acid isomers and benzoic acid, on the determination of color as the result of ester formation from glycerin and phthalic anhydride, on a direct method for determining urea, and on the determination of free and combined formaldehyde. A new study has been started on methods for analysis and specifications for epoxy resins.

*Subcommittee XII on Latex and Emulsion Paints* (P. T. Howard, chairman) is continuing its work on washability and scrubbability with a variation of the abrasive boat technique, on efflorescence with a modified procedure to determine the possible effects of the type of cooling areas used and the role played by circulation of the air, and on freeze-thaw stability with intermittent vibration during continuous freezing at 15 F for 8 days. The exterior exposure work is being continued, and a series of pictorial standards is being collected; the microbiological tests, the coalescence tests, and the package stability tests are also being continued.

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[4] See p. 297.

*Subcommittee XIII on Shellac* (C. C. Hartman, chairman) recommended the immediate adoption of the proposed revisions of the Specifications for Orange Shellac and Other Lacs (D 237 – 55), and of the Methods of Sampling and Analysis of Shellac (D 29 – 40), by the addition to the latter of Sections 6 to 13 on Acid Value of the Tentative Methods of Testing Shellac (D 29 – 54 T). Consideration is also being given to further revision of Standard Method D 29 – 40 as to the desirability of bringing the sampling sections into accord with the current practices of the U. S. Shellac Importers Assn. and the American Bleached Shellac Manufacturers Assn., and with the procedures contained in the International Specifications for Shellac, Seedlac, and Bleached Lac.

*Subcommittee XV on Specifications for Pigments, Dry and in Oil* (C. L. Crockett, chairman) is completing revisions of the Methods of Sampling and Testing Aluminum Powder and Paste (D 480 – 51), and Specifications for Copper Phthalocyanine Blue (D 963 – 49). The subcommittee will undertake the development of specifications for strontium chromate.

*Subcommittee XVI on Printing Inks* (M. C. Rogers, chairman) is completing its work on a proposed method of test for specific gravity of printing inks and is giving further study to tests of other characteristics of printing inks, coverage, ink misting and estimation, and rub resistance.

*Subcommittee XVII on Flash Point* (S. S. Kurtz, Jr., acting chairman) will undertake work on non-viscous materials flashing in the 10 to 50 F range, thereby extending the range of flash point determinations to provide for materials flashing in the range below 50 F as well as those in the range of 60 to 110 F. The latter is already covered by a method. Determination of the flash point of viscous materials of particular interest to the paint industry will also be investigated.

*Subcommittee XVIII on Physical Properties* (E. J. Dunn, Jr., chairman) prepared the proposed Tentative Methods of Test for Oil Absorption of Pigments by the Gardner-Coleman Method, for Knoop Indentation Hardness of Paint, Varnish and Lacquer Coatings, and for Density of Paint, Varnish, Lacquer, and Related Products. The production of uniform films with the wire-wound draw-down bar is being investigated as well as tests for the adhesion of films to metals and the standardization of the No. 2 orifice of the Ford Type Cup. The subcommittee is reviewing the Gardner mobilometer consistency test of Federal Methods of Test TT-P-141, for re-drafting as a new proposed method of test. Water vapor permeability of films and the measurement of film thickness on non-magnetic bases such as wood or plastic are also being investigated.

*Subcommittee XIX on Putty, Glazing and Caulking Compounds* (H. L. Kelfer, chairman) cooperated with Subcommittee VI on Definitions in the preparation of the proposed new tentative definitions for caulking compound and glazing compound and the revision of the definition for putty. Definitions of other terms related to the subcommittee field of interest are in preparation. The subcommittee, in its investigation of accelerated weathering, will evaluate adhesion, flexibility and ultimate appearance including cracking, wrinkling, and both oil spotting and bleeding. Progress has been made in the work on initial and ultimate set and surface dry, on the measurement of slump, and on package stability. In the consideration of workability, emphasis will be placed on the following major factors: handling, ease of knifing or consistency, and pick-up or smoothness of surface left by the knife.

*Subcommittee XXV on Cellulosic Coatings and Related Materials* (N. C. Schultze, chairman) prepared the proposed Tentative Methods of Test for Volatile Matter in Tricresyl Phosphate and for Heptane Miscibility of Lacquer Solvents. It also prepared the proposed revisions of the Tentative Methods of Test for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood (D 1211 – 52 T), for Alcohol in Methyl Isobutyl Ketone (D 1362 – 52 T), and for Permanganate Time of Lacquer Solvents and Diluents (D 1363 – 55 T) and, in cooperation with Subcommittee VII on Accelerated Tests for Protective Coatings, the revision of title and scope of the Tentative Method of Test for Effect of Household Staining Agents on Applied Nitrocellulose Clear and Pigmented Finishes (D 1308 – 54 T), mentioned above. Revision and immediate adoption of the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 53) was also recommended by the subcommittee. Other recommendations included the adoption as standard of Tentative Methods D 1308 and the withdrawal of the Tentative Methods of Test for Ester Value of Tricresyl Phosphate (D 268 – 49 T), and for Purity of Acetone and Methyl Ethyl Ketone (D 1154 – 54 T), neither of which are any longer required by current specifications for lacquer solvents or diluents. The subcommittee also recommended editorial changes in the Method of Test for Color of Clear Liquids (Platinum-Cobalt Scale) (D 1209 – 54), and the Tentative Method of Test for Water in Lacquer Solvents and Diluents (Fischer Reagent Titration Method) (D 1364 – 55 T). Further work is being done on the toluene dilution ratio method and on a method for evaluating perspiration resistance.

*Subcommittee XXIX on Painting of Metals* (A. J. Eickhoff, chairman) is continuing with its work on the classification of ferrous surfaces for painting. It has established new groups to work on the surface preparation of galvanized iron prior to painting, and on the surface preparation of aluminized steel prior to painting.

This report has been submitted to letter ballot of the committee, which consists of 377 voting members, 192 members returned their ballots, of whom 181 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
*Chairman.*

W. A. GLOGER,
*Secretary.*

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EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards a new Tentative Method of Test for Color Difference Using the Color Eye. The new tentative method was accepted by the Standards Committee on December 13, 1957, and it appears in the 1957 Supplement to Book of ASTM Standards, Part 4, bearing the designation D 1495 – 57 T.

## APPENDIX

## RESULTS OF COLLABORATIVE VISCOSITY TESTS

### Report of Subcommittee IX, Group 18 on Viscosity of Transparent Liquids

Prepared by K. E. Holt[1]

*Problem.*—To determine the effect of variations in the internal diameter of the Gardner-Holdt type viscosity tubes on the time of bubble rise—all other factors being equal.

### Experiment Design

Gardner-Holdt type viscosity tubes having the following inside diameters were used: 10.50, 10.65, 10.75, and 10.80 mm. All diameters checked to a tolerance of ±0.025 mm. The tubes were etched with three lines—the first line approximately 28 mm from the bottom of the tube, the second line exactly 73 mm above the first line, and the third line exactly 8 mm above the second line.[2]

Five different standard oils were used to fill the tubes, having viscosities of P, V, Z, Z5, and Z7.[3]

Each diameter tube was filled with each different oil, making 20 tubes to a set:

| Oil | 10.50 mm diam | 10.65 mm diam | 10.75 mm diam | 10.80 mm diam |
|---|---|---|---|---|
| P....... | No. 1 | No. 2 | No. 3 | No. 4 |
| V....... | No. 5 | No. 6 | No. 7 | No. 8 |
| Z....... | No. 9 | No. 10 | No. 11 | No. 12 |
| Z5...... | No. 13 | No. 14 | No. 15 | No. 16 |
| Z7...... | No. 17 | No. 18 | No. 19 | No. 20 |

Six sets of 20 tubes each, as described above, were made up and distributed to the 9 collaborators[4] so that each laboratory checked two different sets. Each laboratory was requested to have two operators run each set twice, preferably having each operator make his determination on two different days.

The viscosities were to be run in a water bath held at 25 ± 0.1 C, reporting the time in seconds (to nearest 0.1 sec) for the bubble to travel the 73 mm between the first and second etched lines.

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[1] Control Director, Archer-Daniels-Midland Co., Minneapolis, Minn.

[2] The tubes as described were manufactured and supplied to the committee at no charge by the R. P. Cargille Co.

[3] These are the standard oils used by the Gardner laboratory in their standard comparator; they were supplied to the committee by the Gardner Laboratory, free of charge.

[4] Mr. Kenneth E. Holt, Archer-Daniels-Midland Co., Minneapolis, Minn.

Mr. Henry A. Ball, Ball Chemical Co., Glenshaw, Pa.

Mr. R. L. Terrill, Spencer Kellogg and Sons, Inc., Buffalo, N. Y.

Mr. J. C. Weaver, Sherwin-Williams Co., Cleveland, Ohio.

Mr. Paul N. Gardner, Gardner Laboratory, Washington, D. C.

Mr. A. F. Bohnert, Enterprise Paint Manufacturing Co., Chicago, Ill.

Mr. Francis Scofield, Nat. Paint, Varnish & Lacquer Assn., Inc., Washington, D. C.

Mr. D. S. Bolley, Baker Castor Oil Co., Bayonne, N. J.

Mr. W. G. Armstrong, E. I. du Pont de Nemours & Co., Inc., Newport, Del.

## DISCUSSION OF RESULTS

*Variations in Tube Diameter:*

The results obtained showed that the smallest diameter tube, tube A (diameter 10.50 mm), gave the highest viscosity reading; tube B (diameter 10.65 mm) the next highest; tube C (diameter 10.75 mm) next highest; with tube D (diameter 10.80 mm) giving the lowest results (see Table IV).

TABLE I.—EFFECT OF TEMPERATURE ON BUBBLE RISE, SEC.

| Tube | 24.4 C | 24.6 C | 24.8 C | 25.0 C | 25.2 C | 25.4 C | 25.6 C |
|---|---|---|---|---|---|---|---|
| No. 2 | 9.6 | 9.5 | 8.9 | 8.8 | 9.2 | 8.8 | 8.5 |
| | ... | 9.5 | 9.1 | ... | ... | 8.9 | 8.5 |
| | ... | ... | ... | ... | ... | 8.9 | ... |
| No. 6 | 9.4 | 9.1 | 9.3 | 9.0 | 8.9 | 8.7 | 8.6 |
| | ... | 9.3 | 9.3 | ... | ... | 8.8 | 8.5 |
| No. 10 | 24.2 | 24.4 | 24.4 | 24.2 | 23.5 | 23.4 | 22.4 |
| No. 14 | 108.3 | 107.8 | 106.3 | 107.2 | 100.3 | 97.5 | 97.9 |

On a percentage basis tube A is 4.78 per cent greater than tube B; tube B is 2.34 per cent greater than tube C; tube C is 2.09 per cent greater than tube D; tube A is 9.48 per cent greater than tube D.

The standard deviations of the results with the different diameter tubes are so nearly equal that the variation is not significant. This variation is even less significant when using only the results of the five laboratories whose results were most consistent:

| | Average Standard Deviation Using All Laboratories | Average Standard Deviation Using 5 Laboratories |
|---|---|---|
| Tube A | 2.09 | 1.00 |
| Tube B | 2.64 | 0.865 |
| Tube C | 1.81 | 1.08 |
| Tube D | 2.79 | 0.802 |

In addition, the coefficient of variation

$$\frac{\text{SD} \times 100}{\text{Average}}$$

shows this very equitable distribution:

| | |
|---|---|
| Tube A | 2.02 |
| Tube B | 2.25 |
| Tube C | 1.95 |
| Tube D | 1.97 |

TABLE II.—EFFECT OF POSITION ON BUBBLE RISE, SEC.

| Tube Position | Tube No. 4 | Tube No. 20 |
|---|---|---|
| Normal | 4.4 | 427.4 |
| 1 Radius Off Line | 4.1 | 381.4 |
| 1 Diameter Off Line | 3.8 | 330.0 |
| Normal | 4.4 | 433.0 |

TABLE III.—STANDARD DEVIATIONS.

| Laboratory | Average Standard Deviation of All Results | Average Standard Deviation, sec | |
|---|---|---|---|
| | | Operator No. 1 | Operator No. 2 |
| [a]1 | 0.768 | 0.865 | 0.593 |
| 2 | 2.23 | 2.17 | 2.24 |
| [a]3 | 0.975 | 0.886 | 0.647 |
| 4 | 1.95 | 1.40 | 1.56 |
| 5 | 2.86 | 1.38 | 4.08 |
| [a]6 | 1.08 | 1.11 | 0.930 |
| [a]7 | 0.678 | 0.669 | 0.691 |
| [a]8 | 1.18 | 1.105 | 1.13 |
| 9 | 3.32 | 4.26 | 7.70 |
| Average of 9 Laboratories | 1.77 | 1.54 | 2.17 |
| [a]Average of 5 Laboratories | 0.936 | 0.927 | 0.798 |

TABLE IV.—AVERAGE TIME OF BUBBLE RISE, SEC.

| Laboratory | Operator Number | P—Viscosity |  |  |  | V—Viscosity |  |  |  | Z—Viscosity |  |  |  | Z5—Viscosity |  |  |  | Z7—Viscosity |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  | Tube A | Tube B | Tube C | Tube D | Tube A | Tube B | Tube C | Tube D | Tube A | Tube B | Tube C | Tube D | Tube A | Tube B | Tube C | Tube D | Tube A | Tube B | Tube C | Tube D |
| I | No. 1 | 4.68 | 4.48 | 4.35 | 4.23 | 9.95 | 9.63 | 9.25 | 9.08 | 25.3 | 24.2 | 23.7 | 23.4 | 111.4 | 107.8 | 104.7 | 101.9 | 464.1 | 442.2 | 430.7 | 421.8 |
|  | No. 2 | 4.55 | 4.38 | 4.35 | 4.18 | 9.58 | 9.13 | 8.90 | 8.68 | 25.2 | 24.2 | 23.5 | 22.9 | 111.9 | 107.2 | 104.1 | 102.1 | 463.5 | 442.4 | 429.7 | 419.9 |
| II | No. 1 | 4.20 | 4.23 | 4.20 | 4.13 | 9.50 | 9.28 | 9.05 | 8.78 | 25.4 | 24.4 | 24.0 | 23.3 | 111.4 | 109.0 | 104.6 | 104.5 | 474.0 | 429.5 | 432.4 | 431.4 |
|  | No. 2 | 4.28 | 4.18 | 4.15 | 4.13 | 9.65 | 9.38 | 9.08 | 8.80 | 25.7 | 24.9 | 24.2 | 23.6 | 110.1 | 109.4 | 103.1 | 103.7 | 473.1 | 447.5 | 427.5 | 424.0 |
| III | No. 1 | 4.73 | 4.58 | 4.33 | 4.18 | 9.70 | 9.30 | 8.98 | 8.85 | 25.5 | 24.5 | 23.9 | 23.3 | 109.5 | 105.2 | 102.0 | 101.0 | 460.3 | 439.4 | 425.0 | 420.2 |
|  | No. 2 | 4.95 | 4.78 | 4.60 | 4.43 | 9.88 | 9.53 | 9.13 | 8.98 | 26.1 | 25.0 | 24.3 | 23.6 | 110.3 | 106.0 | 103.4 | 101.5 | 462.9 | 442.3 | 429.9 | 423.7 |
| IV | No. 1 | 4.87 | 4.57 | 4.49 | 4.34 | 9.96 | 9.60 | 9.28 | 8.92 | 25.9 | 24.4 | 23.8 | 23.5 | 112.2 | 107.5 | 104.0 | 102.6 | 467.3 | 445.1 | 433.9 | 420.4 |
|  | No. 2 | 4.85 | 4.65 | 4.57 | 4.54 | 10.03 | 9.60 | 9.43 | 9.24 | 25.5 | 25.0 | 23.9 | 23.6 | 111.8 | 108.9 | 104.1 | 102.0 | 465.9 | 446.7 | 436.6 | 424.6 |
| V | No. 1 | 4.66 | 4.48 | 4.42 | 4.38 | 9.68 | 9.24 | 8.90 | 8.83 | 24.9 | 23.7 | 23.5 | 23.1 | 112.1 | 107.4 | 103.7 | 101.4 | 461.4 | 441.5 | 424.0 | 391.3 |
|  | No. 2 | 4.73 | 4.45 | 4.43 | 4.18 | 9.55 | 9.20 | 9.08 | 8.78 | 25.5 | 24.4 | 24.2 | 24.2 | 119.3[a] | 112.1 | 102.7 | 101.9 | 451.3 | 420.8 | 418.1 | 396.2 |
| VI | No. 1 | 4.70 | 4.50 | 4.40 | 4.25 | 9.75 | 9.38 | 9.05 | 9.00 | 25.4 | 24.3 | 23.7 | 23.2 | 111.6 | 106.0 | 103.0 | 101.3 | 460.1 | 440.0 | 425.6 | 420.1 |
|  | No. 2 | 4.73 | 4.35 | 4.30 | 4.15 | 9.63 | 9.25 | 9.08 | 8.88 | 25.3 | 24.3 | 23.5 | 23.3 | 111.3 | 106.1 | 103.6 | 101.4 | 462.4 | 442.6 | 431.1 | 422.4 |
| VII | No. 1 | 4.53 | 4.25 | 4.30 | 4.28 | 9.53 | 9.23 | 8.75 | 8.63 | 25.3 | 24.3 | 23.5 | 23.2 | 111.8 | 104.3 | 103.2 | 101.8 | 466.3 | 440.8 | 430.6 | 422.7 |
|  | No. 2 | 4.48 | 4.30 | 4.28 | 4.15 | 9.50 | 9.23 | 8.68 | 8.55 | 25.3 | 24.4 | 23.7 | 23.2 | 111.9 | 105.4 | 103.9 | 102.0 | 466.0 | 441.7 | 430.6 | 423.2 |
| VIII | No. 1 | 4.55 | 4.43 | 4.38 | 4.25 | 9.45 | 9.13 | 8.93 | 8.93 | 25.6 | 24.5 | 23.8 | 23.5 | 112.0 | 107.3 | 102.5 | 102.4 | 474.4 | 448.3 | 440.6 | 431.3 |
|  | No. 2 | 4.70 | 4.35 | 4.23 | 4.23 | 9.48 | 9.18 | 8.80 | 8.93 | 25.7 | 24.6 | 23.7 | 23.4 | 112.1 | 107.0 | 102.2 | 102.6 | 473.8 | 447.6 | 436.2 | 427.6 |
| IX | No. 1 | 4.70 | 4.60 | 4.40 | 4.30 | 9.65 | 8.98 | 9.13 | 9.03 | 24.4 | 23.5 | 22.5 | 22.9 | 110.6 | 109.1 | 105.5 | 103.5 | 443.8 | 454.0 | 446.9 | 449.0 |
|  | No. 2 | 4.88 | 4.70 | 4.60 | 4.75 | 9.53 | 9.53 | 9.10 | 9.18 | 25.3 | 25.0 | 24.1 | 24.3 | 110.8 | 112.3 | 109.2 | 103.3 | 445.6 | 411.0 | 439.1 | 427.7 |
| Range | High | 4.95 | 4.78 | 4.60 | 4.54 | 10.03 | 9.63 | 9.43 | 9.24 | 26.1 | 25.0 | 24.3 | 24.3 | 112.2 | 112.3 | 109.2 | 104.5 | 474.4 | 454.0 | 446.9 | 449.0 |
|  | Low | 4.20 | 4.18 | 4.15 | 4.13 | 9.45 | 8.98 | 8.68 | 8.55 | 24.4 | 23.5 | 22.5 | 22.9 | 109.5 | 104.3 | 102.0 | 101.0 | 443.8 | 411.0 | 418.1 | 391.3 |
| Average |  | 4.65 | 4.46 | 4.38 | 4.25 | 9.67 | 9.32 | 9.03 | 8.89 | 25.4 | 24.4 | 23.75 | 23.4 | 111.3 | 107.7 | 103.9 | 102.3 | 463.1 | 440.2 | 431.6 | 422.1 |
| Standard Deviation |  | 0.195 | 0.163 | 0.123 | 0.104 | 0.177 | 0.148 | 0.188 | 0.179 | 0.370 | 0.402 | 0.404 | 0.357 | 0.803 | 2.182 | 1.61 | 0.944 | 8.89 | 10.28 | 6.73 | 12.38 |

NOTE 1.—Each figure shown is an average of 4 determinations. [a] Not used in calculations.

NOTE 2.—Diameter of tubes as follows: A, 10.50 mm; B, 10.65 mm; C, 10.75 mm; D, 10.80 mm.

This shows that any variation in results can be attributed to the actual difference in diameter of the tubes.

*Variations Between Laboratories:*

The variations between laboratories were quite high and inconsistent. This was due mostly to extremely high variation in four of the laboratories. Standard deviations on all laboratories range from 0.678 to 3.32. Using the same five laboratories whose results were most consistent, the standard deviations range from 0.678 to 1.18. Table III shows the standard deviation for each laboratory.

The fact that the variation in the five laboratories is so small shows that given comparable equipment and using reasonable care in the technique, reproducible results can be obtained between laboratories.

*Operator Variations:*

Table IV gives the actual standard deviations of operators Nos. 1 and 2 of each laboratory.

Again, using the results from the five most consistent laboratories, the variations between operators are so small that this bears out results in the variations between laboratories: good reproducible results can be obtained between operators.

## PROBABLE SOURCES OF ERROR

*Temperature:*

The experiment design specified a water bath capable of maintaining a temperature of 25 ± 0.1 C. Some collaborators reported difficulty in maintaining this temperature, which may account for some of the variable results obtained. To determine the effect of temperature on viscosity, a series of viscosities were run at different temperatures. The results obtained showed that 1 C difference in bath temperature will give approximately a 10 per cent variation in viscosity reading (see Table I).

*Vertical Position of Tubes:*

If the tube is not perfectly vertical while timing the bubble rise, a large error is introduced in the result. Actual checks on viscosities show that if the tube is 1 radius off the vertical, results are 8 to 10 per cent lower. If the tube is 1 diameter off the vertical, results are 20 to 25 per cent lower (see Table II).

*Timer:*

A timer or stop watch with an accuracy of 0.1 sec was required to eliminate errors due to timing. One laboratory reported difficulties with their timing device and applied a correction factor to their results.

*Bubble Size:*

We assumed no variation due to bubble size, as all of the tubes were filled and sealed by one operator.

# REPORT OF COMMITTEE D-2

ON

# PETROLEUM PRODUCTS AND LUBRICANTS*

This report contains the recommendations of the subordinate groups of Committee D-2 on Petroleum Products and Lubricants. The recommendations have been approved by Committee D-2 in accordance with the regulations of the Society, and are presented herewith.

## PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

It is recommended that the following 15 proposed methods and specifications be published as information, as appended hereto[1] or as otherwise indicated:

Appendix I. Optional Method for Analysis of Lubricating Greases.

Appendix II. Particle Size Range of Graphite (Sieve Method).

Appendix III. 20 Deg Specular Gloss of Waxed Paper.

Appendix IV. Specifications for Aviation Turbine Fuels.

Appendix V. Proposed Revisions to ASTM Motor Method D 357 to Permit Rating of Fuels of Over 100 Octane Number.

Appendix V-A. Proposed Revisions to ASTM Research Method D 908 to Permit Rating of Fuels of Over 100 Octane Number.

Appendix V-B. Proposed Revisions of the 1956 Engine Test Manual to Permit Rating of Fuels of Over 100 Octane Number.

Appendix VI. Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene.

Appendix VII. Vanadium in Residual Fuel Oil. (Republished with revisions. See Appendix V to the 1955 Report of Committee D-2.)

Appendix VIII. Thermal Stability of U. S. Navy Special Fuel Oil.

Appendix IX. Carbon Number Distribution of Aromatic Compounds in Naphthas by Mass Spectrometry.

Appendix X. Refractive Index of Viscous Liquids and Melted Solids.

Appendix XI. Distillation of Petroleum Products. (To replace eventually Method D 86, Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products, and Method D 158, Test for Distillation of Gas Oil and Similar Distillate Fuel Oils.)

Appendix XI-A. Specifications for Distillation Equipment.

Appendix XII. Summary of Data Collected in the Cooperative Testing of Method D 240, Test for Heat of Combustion of Liquids by Bomb Calorimeter. (To be appended to Method D 240.)

Appendix XIII. Odor of Petroleum Wax.

Appendix XIV. Supporting Data for Extension of Standard Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity (D 666 - 53) to Permit Conversion of Kinematic to Saybolt Furol Viscosities at 210 F.

Appendix XV. Thermal Stability of Aviation Turbine Fuels.

## NEW TENTATIVES

The committee recommends that the following seven methods be accepted for publication as tentative, as appended hereto[2] or as otherwise indicated:

*Tentative Methods of Test for:*

Low-Temperature Torque of Ball Bearing Greases. (Revision of Appendix IX to the 1956 Report of Committee D-2.)

Measuring the Color of Petroleum Products. (Revision of Appendix XIII to the 1956 Report of Committee D-2.)

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The proposed methods and specifications appear in the compilation of "ASTM Standards on Petroleum Products and Lubricants," November, 1957. The proposed Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene also appears on p. 378.

[2] The new and revised tentatives and standards appear in the 1957 Supplement to Book of ASTM Standards, Part 5.

Freezing Point of Aviation Fuels.

Emulsion Stability of Soluble Cutting Oils. (See Appendix I to the 1955 Report of Committee D-2.)

Density and Specific Gravity of Viscous Materials and Melted Solids by Bingham Pycnometer Method. (See Appendix VII to the 1956 Report of Committee D-2.)

Density and Specific Gravity of Hydrocarbon Liquids by Lipkin Bicapillary Viscous Oil Pycnometer (see Appendix VIII to the 1956 Report of Committee D-2), revised as follows:

*Section 9.*—Change to read as follows:

9. The following data shall be used for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatability.*—Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

| Pycnometer Volume, ml | Repeatability, g per ml |
|---|---|
| 10 | 0.00015 |

(*b*) *Reproducibility.*—Single results submitted by each of two laboratories should be considered suspect if they differ by more than the following amount:

| Pycnometer Volume, ml | Reproducibility, g per ml |
|---|---|
| 10 | 0.00035 |

NOTE 5.—Precision values for pycnometers other than 10 ml in volume have not been obtained through cooperative testing.

Blocking Point of Paraffin Wax (See Appendix III, 1955 Report of Committee D-2), revised as follows:

*New Footnote 4.*—Add a new Footnote 4 to Section 8 to read as follows:

[4] Standard reference waxes in the high and low blocking range are available on request from the following sources:

*High Blocking Range.*—Wax No. 5512, Sun Oil Co., Marcus Hook, Pa., Attention: J. D. Tench.

*Low Blocking Range.*—Wax No. SIS No. 2423 (Esso 5150 Wax), available in 1-lb cans from Esso Research and Engineering Co., Products Research Div., P.O. Box 51, Linden, N. J., Attention: C. A. Neusbaum.

*Section 11.*—Change to read as follows:

11. Results should not differ from the mean by more than the following amounts:

| | |
|---|---|
| (*a*) Picking Point: | |
| Repeatability (one operator and apparatus) | 4 F |
| Reproducibility (different operators and apparatus) | 6 F |
| (*b*) Blocking Point: | |
| Repeatability (one operator and apparatus) | 2 F |
| Reproducibility (different operators and apparatus) | 3 F |

## REVISIONS OF TENTATIVES

The committee recommends that the following 17 tentatives be revised as indicated and continued as tentative:

### D 96 – 52 T,[3] Test for Water and Sediment by Means of the Centrifuge:

*Section 3.*—Change the second sentence to read: "This requires vigorous agitation of the sample immediately be fore transferring the sample to the tube."

*Section 4.*—Change to read as follows:

4. (*a*) Measure exactly 50 ml of industrial 90 benzene (ASTM Specification D 837) into the centrifuge tube and add exactly 50 ml of the oil to be tested. Stopper the tube tightly and shake vigorously until the contents are thoroughly mixed. Immerse the tube to the 100-ml mark for 10 min in the bath maintained at 120 F (49 C).

NOTE 2.—When testing a sample containing waxy material, preheat the oil-benzene mixture to 140 F (60 C) before each whirling; the final temperature of the oil-benzene mixture shall not drop below 115 F (46 C).

(*b*) Shake the tube vigorously for 10 sec, place in a trunnion opposite another tube to establish a balanced condition, and whirl for 10 min at a rate, calculated from the equation in Section 2 (*a*), sufficient to produce a relative centrifugal force (rcf) of between 500 and 700 at the tip of the whirling tubes (see Table III for the relationship between diameter swing, rcf, and rpm). Read and record the combined water and sediment at the bottom of the tube to the nearest 0.05 ml from 0 to 1 ml and to the nearest 0.1 ml above 1 ml. Return the tube to the centrifuge and whirl for 10 min at the same rate. Repeat this operation until the combined volume of water and sediment remains constant for two consecutive readings. In general, not more than two are required.

*Section 5.*—Change to read as follows:

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[3] 1955 Book of ASTM Standards, Part 5.

5. Read the combined total of water and sediment as outlined in Section 4 (*b*), multiply by two, and record the result as the percentage of water and sediment, centrifuge method.

*Section 6.*—Change to read as follows:

6. Results should not differ by more than the following percentages:

| Water and Sediment, per cent | Repeatability (One Operator and One Apparatus) | Reproducibility (Different Operators and Apparatus) |
|---|---|---|
| 0 to 0.5 | 0.1 | 0.2 |
| Over 0.5 to 1.5 | 0.2 | 0.4 |
| Over 1.5 to 3.0 | 0.2 | 0.6 |

### D 381 – 56 T,[4] Test for Existent Gum in Fuels by Jet Evaporation:

The method has been rewritten and revised as appended hereto,[2] in order to include editorial changes and a precision section for the solvent washed gum procedure.

### D 447 – 55 T,[3] Test for Distillation of Plant Spray Oils:

*Note 5.*—Change to read as follows:

NOTE 5.—The calculated correction factors within the temperature range encountered in the distillation, as shown below, will affect the temperature recorded at any given volume by more than 1 F when the barometric pressure varies from 760 mm by more than 10 mm.

### D 808 – 52 T,[3] Test for Chlorine in Lubricating Oils and Greases by the Bomb Method:

*Section 1.*—Add a third sentence to read: "The procedure assumes that compounds containing halogens other than chlorine will not be present."

*Section 5 (e).*—In the first sentence, change "$HNO_3$" to "nitric acid."

Insert the following after the first sentence: "Add an excess of 2 ml of the nitric acid solution."

### D 910 – 56 T,[4] Specifications for Aviation Gasolines:

*Table I.*—Under columns "Grade 80–87," "Grade 91–98," and "Grade 100–130," change the value for net heat of combustion from "18,700" to "18,720."

Under column "Test Method" change the ASTM designation for "Net heat of combustion" from "ASTM D 240" to "ASTM D 1405."

Change Footnote *i* to read as follows:

[i] Use the value calculated from Table I in Method D 1405, Test for Estimation of Net Heat of Combustion of Liquid Petroleum Products. Method D 240, Test for Heat of Combustion of Liquids by Bomb Calorimeter, may be used as an alternative method. In cases of dispute, Method D 240 must be used. In this latter case the minimum values for the net heat of combustion in Btu's per pound shall be 18,700 for Grades 80–87, 91–98, and 100–130.

*Section 9 (e).*—Change to read as follows:

(*e*) *Net Heat of Combustion.*—Method of Test for Estimation of Heat of Combustion of Liquid Petroleum Products (ASTM Designation: D 1405).

*Section 9 (m).*[5]—Delete this paragraph, including Fig. 2, Footnotes 5 and 6, Notes 6, 7, and 8, and replace by the following:

(*m*) *Freezing Point of Gasoline.*—Method of Test for Freezing Point of Aviation Fuels (ASTM Designation: D 1477).

### D 1019 – 56 T,[3] Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates:

*Section 6 (a).*—In the last sentence, change "5 min" to "10 min."

*Section 6 (c).*—In the first sentence change "to stand for 5 min" to "to stand for 10 min."

*New Note 5.*—Add a new Note 5 to

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[4] 1956 Supplement to Book of ASTM Standards, Part 5.

[5] Revision of Section 9 (*m*) was dependent on approval by the Society of the Method of Test for Freezing Point of Aviation Fuels (ASTM Designation: D 1477 – 57 T), see 1957 Supplement to Book of ASTM Standards, Part 5.

the end of Section 5 (*a*), renumbering the present Note 5 and subsequent notes, to read as follows:

NOTE 5.—Pipeting *iso*octane into the flask first and filling to the mark with benzene is specified since benzene expands on dilution with a saturated hydrocarbon. The mixture would contain too little saturated hydrocarbon if the benzene were put in the flask first. Expansion with toluene is less, so this point is not important in preparing test mixture B.

### D 1085 – 56 T,[4] Gaging Petroleum and Petroleum Products:

*New Section 5 (d).*—Add a new Section 5 (*d*) as follows, relettering the subsequent paragraphs accordingly.

(*d*) *Plastic Foam Blanket.*—Some tanks are equipped with a plastic foam blanket, floating on top of the liquid, to reduce evaporation losses. In order to prevent this floating plastic foam from smearing the gage tape and making it difficult to determine the exact liquid level in the tank, a portable gage well is used in conjunction with other standard gaging equipment.

*Section 13* (*c*).—Change to read as follows:

(*c*) *Pressure Lock.*—Lock devices of the pressure type are being developed whereby a tape and bob may be utilized for gaging ship tanks while under pressure.

Several tankers and barges have been equipped with such a lock device which is reported to be rendering satisfactory service. This unit is built to be installed in the present plugholes without any structural change to the vessel or gas-free drydock requirements. The unit fits into the plughole with a synthetic rubber gasket between the unit flange and the plughole lip. A clamp bolts around the body of the unit, having tension studs to pull the flange of the unit down on the gasket between the plughole lip and unit, forming a pressure- and water-tight joint.

(*1*) *Description.*—The lock device consists of a pressure-tight lock with the top cover hinged and fitted with a sight port, a gage reel housing, a foot valve control level, and a window wiper and external handwheel for gage tape operation. The main body extends 18 in. into the tank hatch through the plughole and has a foot valve at the bottom to close off the tank pressure when the top cover is open for changing bobs, thermometers, or taking out samples.

(*2*) *Operation.*—When the lock device is in use, the foot valve is open, permitting observation of the tank bottom or liquid level in a tank which is being filled, without loss of vapor as in the case of an open gaging hatch. All vapors from the tank are emitted through the normal vent system above the deck. Liquid levels are visible through the sight port on the top.

The customary take-bob ullage, samples, temperatures, and water soundings are obtained by inserting equipment into the upper chamber while the foot valve is closed, thereby avoiding escape of vapor. When the top chamber is closed, the foot valve is opened and equipment may be lowered into the tank.

*New Section 18.*—Add a new Section 18 as follows, renumbering subsequent sections accordingly.

18. *Portable Gaging Well.*—A portable gaging well, Fig. 11, made of a copper float and a 7- to 8-in. length of 1-in. copper tube, is necessary when gaging tanks covered with a plastic foam blanket.

*New Fig. 11.*—Include as the new Fig. 11, the Fig. 1 shown in Appendix XV to the 1956 Report of Committee D-2. Renumber all subsequent figures.

*Appendix.*—Replace the present Appendix with the Appendix, "Automatic Gaging Devices," which appears in Appendix XV to the 1956 Report of Committee D-2.

### D 1087 – 52 T,[3] Recommended Practice for Volume Calculations and Corrections in the Measurement of Petroleum and Petroleum Products:

*Section 2.*—Add a new Paragraph (*d*) to read as follows:

(*d*) *Tables.*—All references in this standard to tables followed by a number refer to the ASTM-IP Petroleum Measurement Tables (American Edition) (ASTM Designation: D 1250).

*Section 4* (*c*).—Change the first two sentences to read as follows: Calculate the gross quantity at 60 F by multiplying the measured quantity, corrected for any free water and sediment, by the volume correction factor which corresponds to its gravity. Obtain this factor from Table 6

### TABLE I.

| | *Opening Gage* | *Closing Gage* |
|---|---|---|
| *Calculation:* | | |
| Measured quantity: | | |
| Reference depth | 28 ft 8 in. | 28 ft 8 in. |
| Outage gage | 10 ft 7¾ in. | 20 ft 1⅛ in. |
| Innage gage | 18 ft 0¼ in. | 8 ft 6⅞ in. |
| U. S. gal (capacity table) | 954,238 | 457,982 |
| Free water and sediment: | | |
| Reference depth | 28 ft 8 in. | 28 ft 8 in. |
| Outage gage | 28 ft 5¾ in. | 28 ft 5¾ in. |
| Innage gage | 0 ft 2¼ in. | 0 ft 2¼ in. |
| U. S. gal (capacity table) | 10,020 | 10,020 |
| Measured quantity less FS and W, gal | 944,218 | 447,962 |
| Average temperature, deg Fahr | 50 | 49 |
| Volume correction factor (Table 6) | 1.0049 | 1.0054 |
| *Delivered Gross Quantity:* | | |
| Gross quantity at 60 F, | 944,218 × 1.0049 | 447,962 × 1.0054 |
| or, U. S. gal | 948,845 | 450,381 |
| Subtracting | 450,381 | |
| Delivered gross quantity at 60 F, U. S. gal | 498,464 | |
| *Delivered Net Quantity:* | | |
| Suspended water and sediment: | | |
| From laboratory, per cent | | 1.2 |
| Allowable by contract, per cent | | 1.0 |
| Deductible, per cent | | 0.2 |
| Deductible quantity, U. S. gal | | 997 |
| Net delivered at 60 F: | | |
| U. S. gal (498,464 − 997) | | 497,467 |
| Barrels Calculated (497,467 ÷ 42) | | 11,844.45 |
| Barrels to be Reported | | 11,844 |

### TABLE II.

| | |
|---|---|
| *Roof Resting on Supports at Opening Gage:* | |
| *W*, Weight of floating roof (from capacity table), lb | 126,897 |
| Product gravity at 80 F, deg API | 32.5 |
| Product gravity at 60 F, deg API (Table 5) | 31.1 |
| *P*, Pounds per U. S. gal at 60 F (Table 8) | 7.246 |
| *Q*, Roof displacement (126,897 ÷ 7.246), U. S. gal at 60 F | 17,513 |

| Quantity at 60 F: | *Opening Gage* | *Closing Gage* |
|---|---|---|
| Measured volume, U. S. gal | 103,624 | 715,771 |
| Tank contents temperature, deg Fahr | 81 | 80 |
| Volume correction factor (Table 6) | 0.9910 | 0.9914 |
| Net U. S. gal at 60 F (volume × factor) | 102,691 | 709,615 |
| *Q*, Correction for Roof Displacement, U. S. gal at 60 F. | none | 17,513 |
| Correct net U. S. gal at 60 F | 102,691 | 692,102 |
| Subtracting | | 102,691 |
| Received at 60 F, U. S. gal | | 589,411 |

### TABLE III.

| | *Opening Gage* | *Closing Gage* |
|---|---|---|
| *Calculations:* | | |
| Innage gage | 27 ft 2 in. | 2 ft 1½ in. |
| Water innage | 0 ft 2 in. | 0 ft 2⅛ in. |
| Temperature, deg Fahr | 81 | 80 |
| Gravity at 60 F, deg API | 48.7 | 48.6 |
| Observed gravity at tank temperature, deg API | 50.7 | 50.5 |
| Measured quantity, bbl | 51,043.00 | 3,730.26 |
| Gravity variation from 47 deg API | 3.7 | 3.5 |
| Correction (3.7 × 3.56) bbl | 13.17 | none (roof at rest) |
| Corrected measured quantity, bbl | 51,029.83 | 3,730.26 |
| Volume correction factor (Table 6) | 0.9889 | 0.9894 |
| Quantity at 60 F, bbl | 50,463.40 | 3,690.72 |
| Calculated net quantity at 60 F, bbl | 46,772.68 | |
| Reported net quantity delivered at 60 F, bbl | 46,773 | |

### TABLE IV.

| | |
|---|---|
| *R*, Manometer reading at 70 F, in. | 1¾ |
| Manometer liquid specific gravity 60/60 F | 0.8155 |
| Manometer liquid gravity at 60 F (Table 21), deg API | 42.0 |
| Volume correction factor at 70 F (Table 6) | 0.9952 |
| *M*, Manometer liquid specific gravity 70/60 F (0.8155 × 0.9952) | 0.8116 |
| Product temperature, deg Fahr | 81 |
| Product gravity at 81 F, deg API | 63.8 |
| Product gravity at 60 F, deg API | 61.3 |
| Volume correction factor for product at 81 F (Table 6) | 0.9867 |
| Specific Gravity of product 60/60 F (Table 3) | 0.7339 |
| *P*, Product specific gravity 81/60 F (0.7739 × 0.9867) | 0.7241 |
| *C*, Corrected manometer reading (1.75 × 0.8116 ÷ 0.7241 = 1.961), in. | 2 |
| Innage gage reading | 25 ft 4⅜ in. |
| Corrected innage gage (25 ft 4⅜ in.-2 in.) | 25 ft 2⅜ in. |

### TABLE V.

| | |
|---|---|
| Specific gravity of products 60/60 F | 0.586 |
| Summer filling density (from Tariff No. 8), per cent | 55.5 |
| Temperature of liquid product, deg Fahr | 54 |
| Volume correction factor (Table 34) | 1.006 |
| Shell capacity of car, gal | 11,036 |
| Allowable load at 60 F $\left(11{,}036 \times \frac{0.555}{0.586}\right)$, gal | 10,452 |
| Allowable load at 54 F $\left(\frac{10{,}452}{1.006}\right)$, gal | 10,383 |
| | *Gage, in.* |
| Maximum Loading Gage: | |
| For a capacity table based on innage: | |
| From table at 10,390 gal | 77½ |
| For a capacity table based on outage: | |
| Permissible outage (11,036 − 10,390), gal | 646 |
| From table at 646 gal | 9½ |

of ASTM D 1250; for liquefied petroleum gases, obtain this factor from Table 34."

*Section 4 (f).*—Change the portion of the example covering "Calculations," "Delivered Gross Quantity at 60 F," and the "Delivered Net Quantity at 60 F," as shown in the accompanying Table I.

*New Section 4 (g).*—Add a new Section 4 *(g)* to read as follows:

> (*g*) *Significant Figures.*—In the examples shown in Paragraph (*f*), the "Delivered Net Quantity at 60 F" is reported as 11,844 bbl, whereas the actual figure was calculated to be 11,844.45. The decimal fraction was dropped because such accuracy is beyond the practical limits of measurement.
>
> It is recommended that final quantities should be reduced to five significant figures, except:
>
> (*1*) Where a quantity exceeds a five digit number, the figure should be rounded off to the nearest unit digit.
>
> (*2*) Where smaller quantities are concerned, the figure should not be carried more than two digits beyond the decimal.
>
> *Examples.*—The following are examples: 95.32 tons; 410.72 tons; 999.86 bbl; 1,060.8 tons; 5,108.5 bbl; 75,012 gal; 116,323 bbl; 4,203,486 gal.

*Section 5 (a).*—In the equation delete "$Q = WG$ or."

In the explanation beneath delete "$G$ = gallons per pound of product."

Change the last sentence to read: "Obtain the pounds per gallon of the product from Table 8 of ASTM Standard D 1250."

*Section 5 (b).*—Change the example as shown in the accompanying Table II.

*Section 6 (b).*—Change the portion of the example covering "Calculations" as shown in the accompanying Table III.

*Section 7 (a).*—Change the last sentence to read: "The gravities at the required temperatures may be obtained from Table 6; convert specific gravity of the manometer liquid to API Gravity by use of Table 21."

*Section 7 (b).*—Change the example as shown in the accompanying Table IV.

*Section 9 (a).*—In the first sentence, change "*Freight Tariff 300-F ICC No. A-3739*" to "*Freight Tariff 300-H ICC No. 79.*"

*Section 9 (b).*—In line 12, change "minus" to "–".

Change "Volume correction factor, group 0 at 100 F (Table 7) . . . . . . 0.9861" to "Volume correction factor (Table 6) . . . . . . 0.9853."

Change "Gross quantity at 60 F (10,139 × 0.9861), gal . . . . 9,998" to "Gross quantity at 60 F (10,139 × 0.9853), U. S. gal . . . . 9,990."

*Section 10 (a).*—In the first sentence, change "*Tariff No. 8*" to "*Tariff No. 10.*"

*Section 10 (b).*—Change the example as shown in the accompanying Table V.

TABLE VI.

| Compound | Purity, per cent | Bromine Number | | | | |
|---|---|---|---|---|---|---|
| | | Theory | Color Indicator Method | | Electrometric Method | |
| | | | Found | Deviation | Found | Deviation |
| **Branched Chain Olefins** | | | | | | |
| 2-methyl-trans-3-hexene | 99.9$^a$ | 162.8 | 167.4 | +4.6 | 174.8$^l$ | +12.0 |
| 3-methyl-2-ethyl-1-butene | 99.8$^a$ | 162.8 | 219.2 | +56.4 | 217.9$^m$ | +55.1 |
| 2,5-dimethyl-2-hexene | 99.8$^a$ | 142.4 | 158.0 | +15.6 | 149.1$^n$ | +6.7 |
| **Aromatics, Monocyclic** | | | | | | |
| 1,3-dimethyl-4-ethylbenzene | 99.9$^a$ | 0.0 | 0.3 | +0.3 | 0.0$^o$ | 0.0 |

### D 1158 – 55 T,[3] Test for Bromine Number of Petroleum Distillates (Color-Indicator Method:

*Appendix: Table II.*—Insert the additional listings for Branched Chain Olefins shown in the accompanying Table VI immediately following 2,3,3-trimethyl-1-butene.

Insert the additional listing for Monocyclic Aromatics shown in the accompanying Table VI immediately following 1,3-diethylbenzene (*m*-diethylbenzene).

*Table II.*—Add footnotes *l*, *m*, *n*, and *o* as follows:

[l] In absence of mercuric chloride, the bromine number found was 163.4 (Deviation +0.6).

[m] In absence of mercuric chloride, the bromine number found was 165.4 (Deviation + 2.6).

[n] In absence of mercuric chloride, the bromine number found was 142.8 (Deviation +0.4).

[o] In absence of mercuric chloride, the bromine number found was 0.0 (Deviation 0.0).

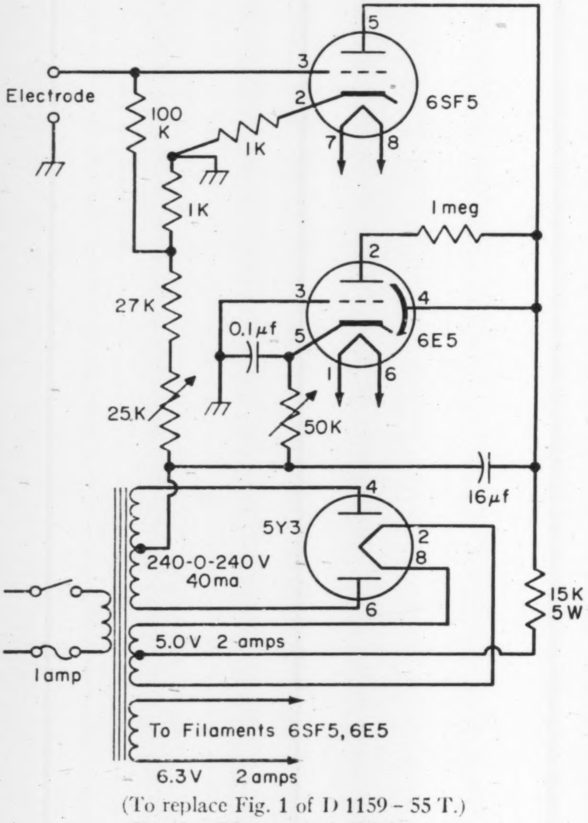
### D 1159 – 55 T,[3] Test for Bromine Number of Petroleum Distillates (Electrometric Method):

*Table II.*—Make the same revision as in Table II of Method D 1158.

*Figure 1.*—Replace with the accompanying Fig. 1.

### D 1160 – 56 T,[4] Test for Reduced Pressure Distillation of Petroleum Products:

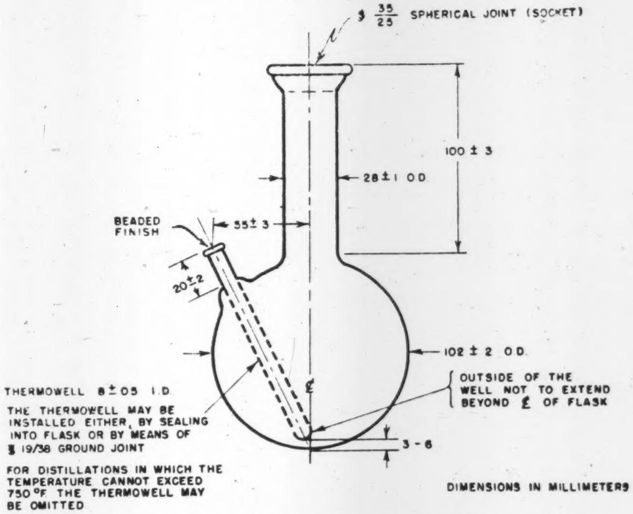
*New Note 2.*—Add the following new Note 2 to the end of Section 2, renumber-


Electrode
100 K
1K
1K
27K
25K
0.1μf
50K
3
2
7
8
5
6SF5
1 meg
2
3
4
5
1
6
6E5
16μf
4
5Y3
2
8
6
240-0-240 V
40 ma
15K
5W
5.0V 2 amps
1 amp
To Filaments 6SF5, 6E5
6.3V 2 amps

(To replace Fig. 1 of D 1159 – 55 T.)

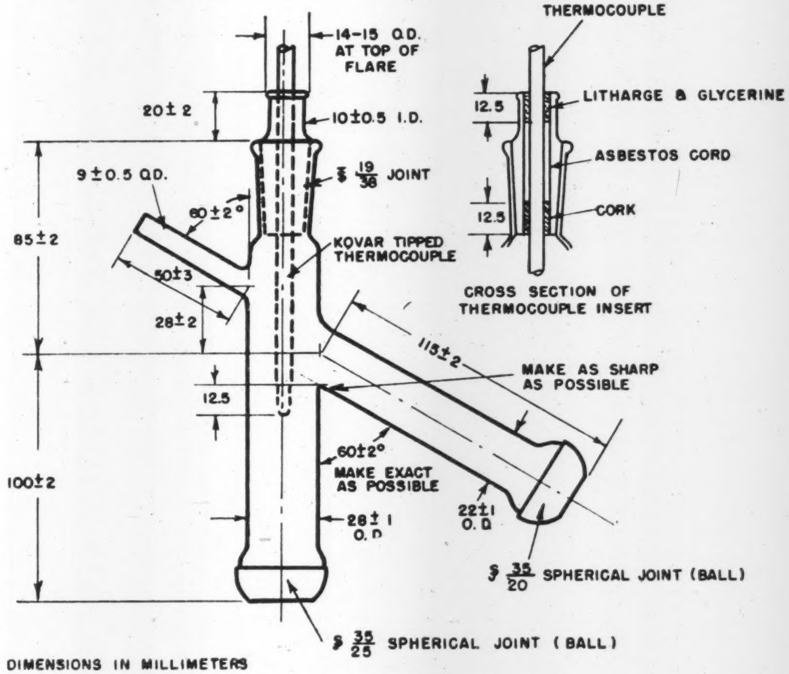
FIG. 1.—Electrometric Titrimeter.

NOTE.—Stancor PC 8401 or equivalent suitable.


§ 35/25 SPHERICAL JOINT (SOCKET)
100 ± 3
28 ± 1 O.D
BEADED FINISH
35 ± 3
20 ± 2
102 ± 2 O.D.
℄
THERMOWELL 8 ± 0.5 I.D
THE THERMOWELL MAY BE INSTALLED EITHER, BY SEALING INTO FLASK OR BY MEANS OF § 19/38 GROUND JOINT
FOR DISTILLATIONS IN WHICH THE TEMPERATURE CANNOT EXCEED 750 °F THE THERMOWELL MAY BE OMITTED
OUTSIDE OF THE WELL NOT TO EXTEND BEYOND ℄ OF FLASK
3 - 6
DIMENSIONS IN MILLIMETERS

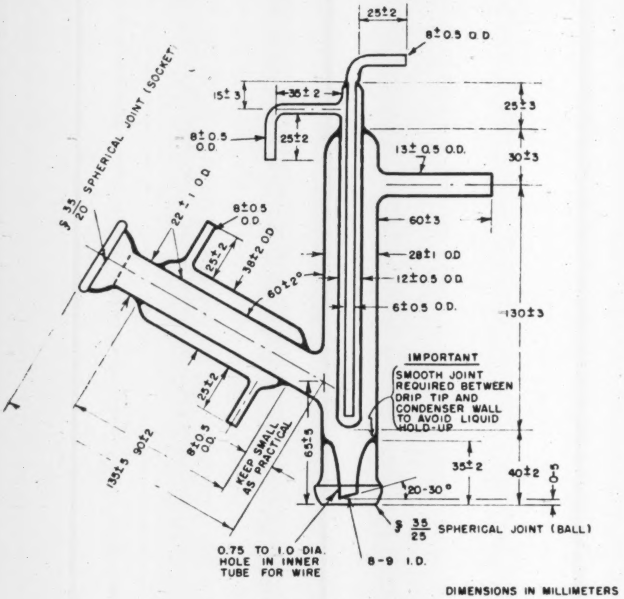
(To replace Fig. 2 of D 1160 – 56 T.)

FIG. 2.—Distillation Flask.


14-15 O.D. AT TOP OF FLARE
20 ± 2
10 ± 0.5 I.D.
§ 19/38 JOINT
9 ± 0.5 O.D.
60 ± 2°
85 ± 2
KOVAR TIPPED THERMOCOUPLE
50 ± 3
28 ± 2
115 ± 2
MAKE AS SHARP AS POSSIBLE
12.5
60 ± 2°
MAKE EXACT AS POSSIBLE
100 ± 2
28 ± 1 O.D
22 ± 1 O.D.
§ 35/20 SPHERICAL JOINT (BALL)
§ 35/25 SPHERICAL JOINT (BALL)
THERMOCOUPLE
12.5
LITHARGE & GLYCERINE
ASBESTOS CORD
12.5
CORK
CROSS SECTION OF THERMOCOUPLE INSERT
DIMENSIONS IN MILLIMETERS

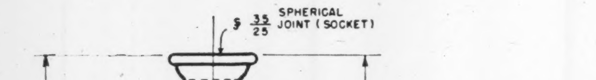
(To replace Fig. 3 of D 1160 – 56 T.)

FIG. 3.—Distillation Column and Thermocouple Installation.


25±2
8±0.5 O.D.
15±3
35±2
25±3
8±0.5 O.D.
25±2
13±0.5 O.D.
30±3
§ 35/20 SPHERICAL JOINT (SOCKET)
22±1 O.D.
8±0.5 O.D.
25±2
38±2 O.D.
60±3
28±1 OD
12±0.5 OD
60±2°
6±0.5 O.D.
130±3
IMPORTANT
SMOOTH JOINT REQUIRED BETWEEN DRIP TIP AND CONDENSER WALL TO AVOID LIQUID HOLD-UP
25±2
8±0.5 O.D.
KEEP SMALL AS PRACTICAL
65±5
90±2
135±5
35±2
40±2
0-5
20-30°
§ 35/25 SPHERICAL JOINT (BALL)
0.75 TO 1.0 DIA. HOLE IN INNER TUBE FOR WIRE
8-9 I.D.
DIMENSIONS IN MILLIMETERS

(To replace Fig. 5 of D 1160 - 56 T.)

FIG. 4.—Condenser.


§ 35/25 SPHERICAL JOINT (SOCKET)
75±5
8±0.5 O.D.
MUST BE ABOVE 200 ML MARK
200ml 20°C TC
180
160
140
120
100
80
60
40
20
25±2
41±1 O.D. (INNER TUBE)
330±5
195±1.5
51 ± 1.5 O.D.
3 INDENTATIONS 120 ° APART TO CENTER TUBE INSIDE AVOID OBSCURING THE GRADUATIONS.
8±0.5 O.D.
NOTE: JACKET CONNECTIONS PLACED SO AS NOT TO INTERFERE WITH READING OF GRADUATIONS
25±2
45±3
DIMENSIONS IN MILLIMETERS

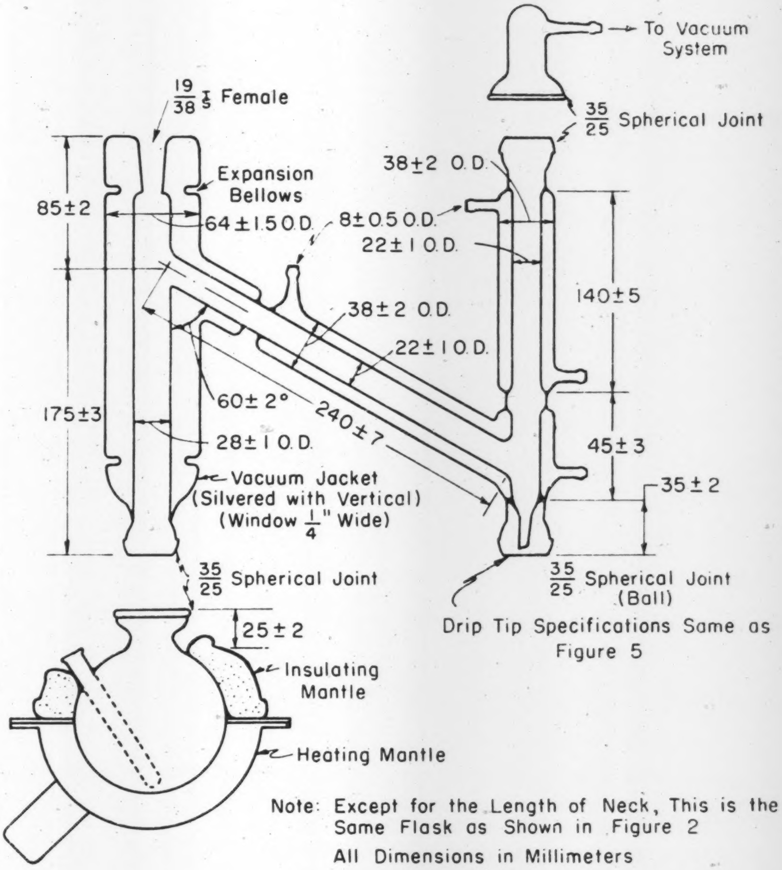
(To replace Fig. 7 of D 1160 - 56 T.)

FIG. 5.—Receiver.

ing the present Note 2 and subsequent notes accordingly:

NOTE 2.—From the data obtained in the application of this method to a particular sample, it may be desired to derive corresponding vapor temperatures at some pressure, up to 760 mm, other than the pressure which was maintained during the test. For this purpose, use may be made of vapor pressure charts[5] for petroleum hydrocarbons. In the use of these charts, the characterization factor, $K$, for the sample shall be assumed to be 12.0, unless another value is assigned to the sample by mutual agreement. Data derived by application of these charts to determined data shall be so designated. It should be realized that the precision as given in Section 8 does not apply to derived data.

*New Footnote 5.*—Add a new Footnote 5 as follows, renumbering the present Footnote 5 and subsequent footnotes:


To Vacuum System
35/25 Spherical Joint
19/38 ₮ Female
38±2 O.D.
Expansion Bellows
85±2
64±1.5 O.D.
8±0.5 O.D.
22±1 O.D.
140±5
38±2 O.D.
22±1 O.D.
60±2°
240±7
175±3
28±1 O.D.
45±3
35±2
Vacuum Jacket (Silvered with Vertical) (Window 1/4" Wide)
35/25 Spherical Joint
35/25 Spherical Joint (Ball)
25±2
Drip Tip Specifications Same as Figure 5
Insulating Mantle
Heating Mantle
Note: Except for the Length of Neck, This is the Same Flask as Shown in Figure 2
All Dimensions in Millimeters

(To replace Fig. 10 of D 1160 - 56 T.)

FIG. 6.—Vacuum-Jacketed Column Assembly and Heating Mantle.

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[5] J. B. Maxwell and L. S. Bonnell, "Derivation and Precision of a New Vapor Pressure Correlation for Petroleum Hydrocarbons," *Industrial and Engineering Chemistry*, Vol. 49, pp. 1187–1196 (1957).

Charts for this purpose are available upon request to W. T. Gunn, Secretary of Committee D-2, Room 2040, 50 West 50th St., New York 20, N. Y.

*Appendix: Figures 2, 3, 5, 7, and 10.*—Replace Figs. 2, 3, 5, 7, and 10 with the accompanying Figs. 2, 3, 4, 5, and 6.

*Figure 4.*—Delete the 75-mm dimension and the dimension lines.

*Section A1 (d).*—Add a second sentence to read: "The graduated portion of the receiver shall be graduated from the bottom to contain, total volume 200 ml, ring every 20 ml, long line every 10 ml, short line every 2 ml, tolerance ±1.4 ml."

**D 1220 – 56 T,[6] Calibrating Liquid Containers (Upright Tanks):**

*Title.*—Change to read: "Calibrating Upright Tanks."

*Section 13.*—Add a new Paragraph (*c*) to read as follows:

(*c*) Vertical or upright tanks should be remeasured and recalibrated:

(*1*) When restored to service after being disconnected or abandoned.

(*2*) When disassembled and re-erected, or when "moved bodily."

(*3*) When deadwood is changed, or when concrete or other material is placed on the bottom or on the shell of the tank, or when the tank is changed in any manner which would affect the incremental or total volume.

*Section 52.*—Delete Subparagraphs (*1*), (*2*), and (*3*) and substitute the following:

(*1*) For non-insulated metal tanks, the temperature of the shell may, in general, be taken as the mean of the adjacent liquid and ambient temperatures on opposite sides at the same location. In which case:

$$\frac{t_S = t_L + t_A}{2} \quad \ldots\ldots\ldots\ldots (9)$$

(*2*) For insulated metal tanks, the temperature of the shell may be taken as closely apapproximating the adjacent liquid temperature. In which case, $t_S = t_L$.

Volume correction, per cent = $0.0013\ (t_S - 60)$

In applying these principles to upright cylindrical steel tanks, the horizontal cross-sectional area may be taken as a function of tank calibration. The coefficient 0.0013 is predicated on a thermal coefficient 0.0000065[6] per degree Fahrenheit for expansion or contraction. The height (depth) dimension may be taken as a function of gaging the tank for liquid level, predicated on the steel thermal change factor of 0.0000065[6] per degree Fahrenheit. There should be consideration of the corresponding effects which may be present for the steel gaging tape, differentiating between innage and outage measurements.

In applying those principles to horizontal steel tanks and other forms of containers such as spherical or spheroidal tanks, it may be necessary to take all three dimensions of length, width, and depth as functions of tank calibration, using the applicable metal thermal change factor. The application of the principles to any type or form of tank is dependent on the desires of the tank user and the recommendations made in applicable sections of the tank calibration manual.

*New Footnote 6.*—Add a new Footnote 6 as follows, renumbering the present Footnote 6 and subsequent footnotes.

[6] Applies only to mild steel. Appropriate factor to be used for other metal.

*Table II.*—Under "Critical Measurements," opposite "Welded Steel, One or More Rings," delete "and 80." Also, add the superscript "*d*" to follow the first sentence under "Critical Measurements."

*Table II: New Footnote "d."*—Add a new Footnote "*d*" to read as follows:

(*d*) For one-ring tanks, two circumferential measurements shall be taken at 20 per cent and 80 per cent down from the top of the ring. For tanks of more than one ring, if obstructions block the tape path at the 20 per cent down plane the measurement may be taken at a point 80 per cent down. If circumference measurements taken on successive rings indicate unusual variations or distortions, sufficient additional measurements should be taken to satisfy the requirements of all concerned.

*Figure 7-A.*—For both butt-welded and lap-welded illustrations, delete the lower horizontal long arrow on each ring which points to the 80 per cent down point.

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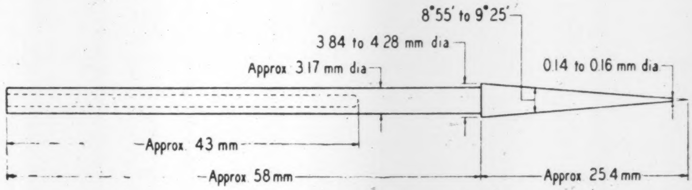
[6] ASTM Manual on Calibrating Liquid Containers (Upright Tanks), January, 1954. Issued as a separate publication.

*Figure 11.*—Delete the letters and arrows labeled "A, C, E, G." Reletter "B, D, F, H" to "A, B, C, D," respectively.

Change the sentence under "*For Critical Measurements:*" to read: "A circumference measurement should be made on each ring at a point located 20 per cent of the exposed ring height below the circumferential weld on the outer surface."

### D 1266 – 55 T,[3] Test for Sulfur in Petroleum Products and Liquefied Petroleum Gases by the $CO_2$-$O_2$ Lamp Method:

Revise as appended hereto.[2]



Shaft drilled out and length adjusted to give final weight of 2 5 ± 0 05 grams

(To replace Fig. 2 of D 1321 – 55 T.)

FIG. 7.—Standard Needle.

### D 1317 – 54 T,[3] Test for Chlorine in Lubricating Oil (Sodium Alcoholate Volumetric Method):

*Section 1.*—Change the last sentence to read: "The procedure assumes that compounds containing halogens other than chlorine will not be present."

*Section 4 (d).*—Change to read as follows:

(*d*) *Nitric Acid* (*approximately 12 per cent by weight*).—Dilute 127 ml of $HNO_3$ (sp gr 1.42) to 1 liter with water. Aerate to remove oxides of nitrogen.

*New Section 4 (i).*—Add a new Section 4 (*i*) as follows, relettering the present Paragraph (*i*) as (*j*):

(*i*) *Sodium Hydroxide* (*approximately 7.5 per cent by weight*).—Dissolve 80 g of NaOH in water and dilute to 1 liter.

*Section 5 (c).*—Change the second and third sentences to read: "Wash the flask with 50 ml of 12 per cent nitric acid and then with 50 ml of naphtha. Add these washings to the separatory funnel. Adjust the acidity of the aqueous layer until just acid to phenolphthalein, using 12 per cent nitric acid and 7.5 per cent sodium hydroxide reagents for this purpose; then add 12 to 13 ml of 12 per cent nitric acid."

*Section 5 (e).*—Change the second and third sentences to read: "If the solution is clear, proceed with the directions in Paragraphs (*f*) and (*g*); if not, filter the solution through a qualitative filter paper into another 500-ml glass-stoppered Erlenmeyer flask. Rinse the original flask with two 25-ml portions of distilled water using each portion to wash the filter paper."

### D 1320 – 54 T,[3] Test for Tensile Strength of Paraffin Wax:

*Footnote 5.*—Change to read as follows:

[5] A solution of 2 per cent Dow-Corning No. 200 Fluid (1000 cs at 25 C) in *iso*octane.

*Section 6 (a).*—Change the last sentence to read: "Apply a very thin film by wiping with a cotton swab saturated with the solution, and allow the solvent to evaporate."

### D 1321 – 55 T,[3] Test for Needle Penetration of Petroleum Waxes:

*Section 1.*—In the second sentence change the number "50" to "100."

*Section 4 (c).*—Change the first sentence to read: "The needle shall be made from a cylindrical stainless steel rod approximately 83 mm long and 4.3 mm in diameter conforming to the requirements given in Fig. 2."

*Section 4 (g).*—In the first sentence, change "$2\frac{1}{8}$ in." to "$2\frac{1}{2}$ in."

Replace the last sentence with the following: "At temperatures higher than 77 F, heat losses may cause the test bath temperature to be lower than the water bath temperature. In these cases, the test bath should be controlled at the test temperature, and an alternate water bath should be used to condition the test samples."

*Figure 2.*—Replace with the accompanying Fig. 7.

*Section 4 (h).*—Replace the last sentence with the following:

> The thermometer shall be immersed to the 70 F mark, and emergent stem correction shall be made as specified in Method E 77, Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers. For higher temperatures, other appropriate ASTM thermometers, such as 18F, 19F, or 64F, should be used in both the water bath and the test bath. The thermometers should be immersed at least 3 in. and emergent stem corrections applied. In such cases, the type of thermometer used shall be included in the test report. The thermometer in the test bath may be mounted at an angle to obtain sufficient immersion. The thermometer bulb should not touch the bottom of the test bath.

*Note 5.*—Change the number "50" to "100."

*Section 6 (c).*—Change the second sentence to read: "Raise the water level in the test bath until the cone of the penetration needle is completely covered."

*Section 8.*—Change to read as follows:

> 8. The following data should be used for judging the acceptability of results (95 per cent probability):

| | **Repeatability** Same Operator and Apparatus | **Reproducibility** Different Operators and Apparatus |
|---|---|---|
| 0 to 30 | 2 | 4 |
| 30 to 50 | 4 | 8 |
| 50 to 100 | 6 | 17 |

### D 1405 – 56 T,[4] Test for Estimation of Heat of Combustion of Liquid Petroleum Products:

*Title.*—Change to read: "Estimation of Net Heat of Combustion of Liquid Petroleum Products."

*Note 2.*—Change the table to read as follows:

| | |
|---|---|
| MIL-F-5572 | Aircraft Engine Fuel (Grades 100/130 and 115/145 |
| MIL-F-5624 | Aircraft Turbine and Jet Engine Fuel (JP-3 and JP-4) |
| MIL-F-7914 | Aircraft Turbine and Jet Engine Fuel (JP-5) |

*Footnote 4.*—Change to read as follows:

> [4] R. S. Jessup and J. A. Cogliano, "Relation Between Net Heat of Combusion and Aniline-Gravity Product of Aircraft Fuels," ASTM BULLETIN, No. 201, October, 1954, p. 55 (TP 217); also the National Bureau of Standards findings as reported by T. W. Armstrong, R. S. Jessup, and T. W. Mears, "Net Heat of Combustion of Aviation Gasoline and Its Correlation with Composition and Aniline-Gravity Product," Wright Air Development Center Technical Report No. 56-504, July, 1956.

*Section 2 (a).*—Change table to read as follows:

| Type Fuel | Equation | Equation No. |
|---|---|---|
| Aviation Gasoline, Grades 100–130 and 115–145 | $Q_p$ (net) = 18 037 + 0.0883($A \times G$) | (1) |
| JP-3 | $Q_p$ (net) = 17 940 + 0.1056($A \times G$) | (1A) |
| JP-4 | $Q_p$ (net) = 17 977 + 0.1056($A \times G$) | (2) |
| JP-5 | $Q_p$ (net) = 17 914 + 0.1056($A \times G$) | (3) |

*Section 2 (c).*—In the first sentence, change "1, 2," to "1, 1A, 2," and delete the number "121."

*Section 4 (b).*—In the first sentence, change "I, II," to "I, IA, II."

In the second sentence, change "Table I should be used for Aviation Gasolines

TABLE VII.—VALUES OF $Q_o$ (NET) FOR AVIATION GASOLINES CALCULATED FROM EQS 1 AND 4.

(New Table I for Method D 1405 – 56 T)

| Aniline-Gravity Product | Net Heat of Combustion, Btu per lb | | | | |
|---|---|---|---|---|---|
| | 0 per cent Sulfur | 0.1 per cent Sulfur | 0.2 per cent Sulfur | 0.3 per cent Sulfur | 0.4 per cent Sulfur |
| 4 000 | 18 391 | 18 377 | 18 363 | 18 349 | 18 335 |
| 4 200 | 18 409 | 18 395 | 18 381 | 18 367 | 18 352 |
| 4 400 | 18 426 | 18 412 | 18 398 | 18 384 | 18 370 |
| 4 600 | 18 444 | 18 430 | 18 416 | 18 402 | 18 388 |
| 4 800 | 18 462 | 18 448 | 18 433 | 18 419 | 18 405 |
| 5 000 | 18 479 | 18 465 | 18 451 | 18 437 | 18 423 |
| 5 200 | 18 497 | 18 483 | 18 469 | 18 455 | 18 440 |
| 5 400 | 18 515 | 18 500 | 18 486 | 18 472 | 18 458 |
| 5 600 | 18 532 | 18 518 | 18 504 | 18 490 | 18 476 |
| 5 800 | 18 550 | 18 536 | 18 522 | 18 507 | 18 493 |
| 6 000 | 18 568 | 18 553 | 18 539 | 18 525 | 18 511 |
| 6 200 | 18 585 | 18 571 | 18 557 | 18 543 | 18 528 |
| 6 400 | 18 603 | 18 589 | 18 574 | 18 560 | 18 546 |
| 6 600 | 18 621 | 18 606 | 18 592 | 18 578 | 18 564 |
| 6 800 | 18 638 | 18 624 | 18 610 | 18 595 | 18 581 |
| 7 000 | 18 656 | 18 642 | 18 627 | 18 613 | 18 599 |
| 7 200 | 18 674 | 18 659 | 18 645 | 18 631 | 18 616 |
| 7 400 | 18 691 | 18 677 | 18 663 | 18 648 | 18 634 |
| 7 600 | 18 709 | 18 695 | 18 680 | 18 666 | 18 652 |
| 7 800 | 18 727 | 18 712 | 18 698 | 18 684 | 18 669 |
| 8 000 | 18 744 | 18 730 | 18 716 | 18 701 | 18 687 |
| 8 200 | 18 762 | 18 748 | 18 733 | 18 719 | 18 704 |
| 8 400 | 18 780 | 18 765 | 18 751 | 18 736 | 18 722 |
| 8 600 | 18 797 | 18 783 | 18 768 | 18 754 | 18 740 |
| 8 800 | 18 815 | 18 800 | 18 786 | 18 772 | 18 757 |
| 9 000 | 18 833 | 18 818 | 18 804 | 18 789 | 18 775 |
| 9 200 | 18 850 | 18 836 | 18 821 | 18 807 | 18 792 |
| 9 400 | 18 868 | 18 853 | 18 839 | 18 824 | 18 810 |
| 9 600 | 18 886 | 18 871 | 18 857 | 18 842 | 18 828 |
| 9 800 | 18 903 | 18 889 | 18 874 | 18 860 | 18 845 |
| 10 000 | 18 921 | 18 906 | 18 892 | 18 877 | 18 863 |
| 10 200 | 18 939 | 18 924 | 18 909 | 18 895 | 18 880 |
| 10 400 | 18 956 | 18 942 | 18 927 | 18 912 | 18 898 |
| 10 600 | 18 974 | 18 959 | 18 945 | 18 930 | 18 915 |
| 10 800 | 18 992 | 18 977 | 18 962 | 18 948 | 18 933 |
| 11 000 | 19 009 | 18 995 | 18 980 | 18 965 | 18 951 |
| 11 200 | 19 027 | 19 012 | 18 998 | 18 983 | 18 968 |
| 11 400 | 19 045 | 19 030 | 19 015 | 19 001 | 18 986 |
| 11 600 | 19 062 | 19 048 | 19 033 | 19 018 | 19 003 |
| 11 800 | 19 080 | 19 065 | 19 050 | 19 036 | 19 021 |
| 12 000 | 19 098 | 19 083 | 19 068 | 19 053 | 19 039 |

and JP-3 jet type fuels" to "Table I should be used for Aviation Gasolines; Table IA for JP-3 jet-type fuels;".

*New Table I.*—Add, as new Table I, the accompanying Table VII.

*Table I.*—Renumber Table I as Table IA and change the title to read: "Table IA.—Values of $Q_p$ (net) for JP-3 Fuels Calculated From Equations 1A and 4."

### REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

**D 240 – 50,[3] Test for Heat of Combustion of Liquids by Bomb Calorimeter:** Revise as appended hereto.[2]

**D 873 – 49,[3] Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method):** Revise as appended hereto.[2]

**D 874 – 55,[3] Test for Sulfated Residue from New Lubricating Oils:** Revise as appended hereto.[2]

### WITHDRAWAL OF TENTATIVES

The committee recommends withdrawal of the following tentatives:

**D 90 – 55 T,[3]** Test for Sulfur in Petroleum Products by the Lamp-Gravimetric Method,
**D 976 – 48 T,[3]** Test for Color of U. S. Military Gasoline, Automotive (Combat), by Means of an ASTM Color Standard.

### REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends immediate adoption of revisions to the eight standards listed below and accordingly asks for a favorable nine-tenths vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

**D 87 – 42,[3] Test for Melting Point of Paraffin Wax:**

The method has been rewritten and revised as appended hereto.[2] The stirring step has been eliminated.

**D 92 – 56,[4] Test for Flash and Fire Points by Cleveland Open Cup:**

*Note 1.*—Add the following after the last sentence: "ASTM Method D 1310, Test for Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus, may be employed if the flash point is below 175 F (79 C) as determined by Method D 92."

**D 97 – 47,[3] Test for Cloud and Pour Points:**

*Section 3* (*g*).—Renumber as Note 1 the present note following this Section.

*Section 4* (*a*).—In the second sentence, add the word "lintless" between "dry" and "filter paper."

*Section 4* (*g*).—Insert the following Note 2 at the end of Section 4 (*g*):

NOTE 2.—A wax cloud or haze is always noted first at the bottom of the test jar where the temperature is lowest. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature is lowered, is usually due to traces of water in the oil. Generally, this water haze will not interfere with the determination of the wax cloud point. In most cases of interference, filtration through dry lintless filter papers such as described in Paragraph 4 (*a*) is sufficient.

In the case of diesel fuels, however, if the haze is very dense, a fresh portion of the sample should be dried by shaking 100 ml with 5 g of anhydrous sodium sulfate for at least 5 min and then filtering through dry lintless filter paper. Given sufficient contact time, this will remove or sufficiently reduce the water haze so that the wax cloud can be readily discerned. Drying and filtering should be done always at a temperature at least 25 F above the approximate cloud point but otherwise not in excess of 120 F. Remove moisture, if present, by any suitable method, as by filtration through dry lintless filter paper until the oil is perfectly clear, but make such filtration at a temperature at least 25 F above the approximate cold point.

**D 128 – 47,[3] Analysis of Lubricating Grease:**

*Section 1* (*b*).—Change the first

sentence to read: "These methods are not suitable for greases made with non-soap thickeners, or with oily constituents other than petroleum oil."

*Section 3 (a)*.—Change to read as follows:

3. (*a*) *Normal Hexane, Commercial Grade*[3] shall conform to the following requirements:

| Test | Requirement | ASTM Designation |
|---|---|---|
| Initial boiling point, min, deg Fahr | 150 | D 216 |
| Dry point, max, deg Fahr | 156 | D 216 |
| Nonvolatile matter, max, per 100 ml | 0.001 | D 268 |
| Color, Saybolt, min | +30 | D 156 |
| Reaction with alkalies | [a] | |

[a] Boil 125 ml of *n*-hexane with 10 ml of 0.5 *N* alcoholic KOH and 50 ml of neutral 50 per cent alcohol for 1½ hr on a hot plate, using a glass tube about 7 mm in internal diameter and 75 cm in length as a reflux condenser. After cooling, titrate the solutions with 0.5 *N* HCl and phenolphthalein. Not less than 9.8 ml of 0.5 *N* HCl shall be required for neutralization. The amount of alkali consumed in this test shall be deducted as a blank correction in the fat determination on solution *E*.[4]

*New Footnote 3*.—Add a new Footnote 3 as follows, renumbering the present Footnotes 3, 4, and 5 as Footnotes 4, 5, and 6.

[3] Normal Hexane is available from Phillips Petroleum Co., Special Products Division, Bartlesville, Okla.

*Figure 1*.—Change all references to "G.A.N." or "Grease Analysis Naphtha" to "*n*-hexane."

*Section 3 (e)*.—Delete Paragraph (*e*) and reletter the following paragraphs accordingly.

*Section 10*.—Change the last two sentences to read as follows:

If undissolved mineral matter or other filler is present, filter both layers while warm through a Gooch crucible provided with a suitable mat, wash the beaker and crucible with warm (140 to 145 F)[7] water and warm *n*-hexane, and finally wash the crucible with alcohol, collecting the alcohol washings separately and discarding them. Dry the crucible and contents at 248 F and weigh, reporting the result as "percentage of insoluble matter" (graphite, mica, talc, asbestos, gypsum, wood pulp, etc.).

*New Footnote 7*.—Add a new Footnote 7 as follows, renumbering the present Footnotes 6, 7, 8, and 9 as Footnotes 8, 9, 10, and 11.

[7] Throughout the method where the word "warm" is used, it is to be understood that a temperature of 140 to 145 F is to be used.

*Section 11*.—Change to read as follows:

11. If gypsum is present as a filler, the results obtained in the determination described in Section 10 will be too low because of the solubility of $CaSO_4$ in HCl. In this case, weigh a sample of about 5 g of grease in a small beaker, and decompose with 50 ml of *n*-hexane and 25 ml of concentrated HCl, warming on the steam bath until all gypsum is dissolved. Pour the contents of the beaker into a separatory funnel, rinse the beaker with warm water and a little warm *n*-hexane (**Caution**, see Note 1). and allow the solutions in the funnel to separate clearly. Draw the lower layer into a beaker, wash the *n*-hexane in the separator with two portions of 20 ml each of warm HCl (10 per cent), and add the washes to the strong acid solution in the beaker. Evaporate the HCl solution in the beaker on the steam bath almost to dryness, dilute to 150 ml with water, heat to boiling, and treat with 10 ml of $BaCl_2$ solution (10 per cent). Then filter off the $BaSO_4$ and ignite in the usual way, calculate the percentage of $CaSO_4$ from the weight of the $BaSO_4$, and report it as $CaSO_4 \cdot 2H_2O$.

*Note 1*.—**Caution**.—Extreme care should be exercised in handling warm *n*-hexane because of pressure developing in the stoppered separatory funnel. Invert the funnel and release pressure through the stopcock occasionally.

*Note 2*.—Chalk and other forms of alkaline earth carbonates are not determined by this method, but are considered under the determination of free alkalinity, Section 20.

*Section 12 (a) and (b)*.—Change to read as follows:

12. *Procedure*.—(*a*) If fillters are absent, weigh the grease sample (Section 2) into either a 250-ml or a 500-ml Erlenmeyer flask, spreading

the sample over the lower inside surfaces of the flask. Add 20 ml of *n*-hexane followed by 50 ml of HCl (10 per cent) and several boiling chips. Digest the sample by boiling under a reflux condenser until it is completely decomposed. Formation of a clear oil layer is evidence of complete decomposition. Three hours' digestion time is generally sufficient; however, more vigorous boiling or use of a stronger HCl solution or additional *n*-hexane will be found necessary for greases difficult to decompose. Allow the contents of the flask to cool to room temperature. If contents remain liquid, follow the procedure prescribed in Paragraph (*b*). If contents congeal or if solid particles form, follow the procedure prescribed in Paragraph (*c*).

(*b*) Transfer the contents of the flask to a separator, using *n*-hexane and distilled water as washing liquids. Allow this solution (or the combined filtrate and washings, except the alcohol washings, from the determination of fillers, Section 10, transferred to a separator), to clear, and draw the aqueous layer, *A*, which contains all the bases as well as glycerin, into another separator. Wash the *n*-hexane layer, *B*, three times with 25 ml of water to remove HCl, adding the washings to *A*. Wash the aqueous solution, *A*, twice with 20 ml of *n*-hexane, *C*, and then set solution *A* aside for examination for glycerin (Section 14). Wash solution *C* once with 15 ml of water, which may then be discared, and add *C* to *B*. If solutions *B* and *C* are comparatively light-colored, an approximate determination of free fatty acids and fatty acids from soap may now be made by titrating the solution in the separator with 0.5 *N* alcoholic KOH and phenolphthalein, using 200 as the average neutralization value of the fatty acid (that is, 1.0 g of fatty acid requires 200 mg of absolute KOH for neutralization). If solutions *B* and *C* are dark, add a few drops of phenolphthalein and sufficient 0.5 *N* alcoholic KOH to make the alcoholic layer distinctly alkaline after vigorous shaking.

(*c*) Heat the contents of the flask to approximately 145 F, and transfer to a separator that has been previously warmed by flushing with warm water, using warm *n*-hexane and warm distilled water as washing liquids (**Caution,** see Note 1 of Section 11). Allow this solution to clear, and draw the aqueous layer, *A*, which contains all the bases as well as glycerin, into another separator. Wash the *n*-hexane layer, *B*, three times with 25 ml of warm water to remove HCl, adding the washings to *A*. Wash the aqueous solution, *A*, twice with 20 ml of warm *n*-hexane, *C*, and then set solution *A* aside for examination for glycerin (Section 14). Wash solution *C* once with 15 ml of warm water, which may then be discarded, and add *C* to *B*. If solution *B* and *C* are comparatlively light-colored, an approximate determination of free fatty acids and fatty acids from soap may now be made by titrating the solution, which is at approximately 145 F, in the separator with 0.5 *N* alcoholic KOH and phenolphthalein, using 200 as the average neutralization value of the fatty acid (that is, 1.0 g of fatty acid requires 200 mg of absolute KOH for neutralization). If solutions *B* and *C* are dark, add a few drops of phenolphthalein and sufficient 0.5 *N* alcoholic KOH to make the alcoholic layer distinctly alkaline after vigorous shaking.

(*d*) If the solutions *B* and *C* from Paragraphs (*b*) and (*c*) have been titrated, add 0.5 to 1.0 ml excess of alkali before separating. The conservative addition of alcohol at this point may aid in securing rapid and sharp separation. Allow the two solutions to separate sharply and draw off the lower alcoholic layer, *D*, into another separator. Wash the upper *n*-hexane layer, *E*, three times, with 30, 25, and 20 ml of neutral 50 per cent alcohol, respectively, adding these washes to *D*. Wash solution *D* with 25 ml of *n*-hexane after which draw off *D* into a beaker and add the *n*-hexane to *E*. Evaporate solution *D* to a small volume to remove alcohol, wash the residue of potassium soap into a separator with hot water, acidify with HCl, and shake out twice with 50 and 25 ml of ethyl ether, *F*, respectively. Run solution *F* into another separator, and wash twice with 20 ml portions of water, which may then be discarded. Transfer solution *F* to a weighed beaker and evaporate to dryness on the steam bath, blowing with air to remove all traces of ethyl ether. Heat the residue, consisting of free fatty acids and fatty acids from soap, for a short time on a steam bath, adding and evaporating 5-ml portions of acetone until a constant weight is obtained and the last traces of water are removed. The exact neutralization value shall then be determined on as large a sample of these fatty acids as possible.

*Section 12 (c)*.—Reletter as Section 12 (*e*).

*Section 12 (d)*.—Reletter as Section 12 (*f*) and change the reference to "grease analysis naphtha" to *n*-hexane."

*Sections 15 (a) and (b), 16 (a), 17, and 19*.—Change all references to "grease analysis naphtha" or "grease analysis naphtha solution" to "*n*-hexane."

*Section 18.*—In line 3, change "120 C" to "248 F."

*Section 29 (a).*—Change the first and second sentences to read as follows:

To determine free glycerin in grease, the sample is refluxed with dilute sulfuric acid and *n*-hexane. The *n*-hexane is evaporated to reduce subsequent emulsion difficulties, the warm mixture extracted with warm water, the washings neutralized and oxidized with potassium periodate,[10] and the resulting solution titrated with standard sodium hydroxide solution.

*Section 30 (a).*—Change "benzene" to "*n*-hexane."

*Section 30 (b).*—Change to read as follows:

(*b*) Reflux the mixture on a steam bath until the grease is disintegrated. Allow the contents of the flask to cool to room temperature. If contents remain liquid, follow the procedure prescribed in Paragraph (*c*). If contents congeal or if solid particles form, follow the procedure prescribed in Paragraph (*d*).

*Present Section 30 (d).*—Reletter as Paragraph (*e*) and change the reference to "grease analysis naphtha solutions" to "*n*-hexane."

*New Section 30 (d).*—Add a new Paragraph (*d*) as follows, relettering the subsequent paragraphs accordingly:

(*d*) Heat contents of the flask to approximately 145 F and transfer quantitatively to a 500-ml separator, washing the flask with 50 ml of warm *n*-hexane and 25 ml of warm distilled water, and introducing these washings into the separator. Keep the contents of the separator warm, shake, and allow the two layers to separate. Draw off the water layer into a second separator contining 100 ml of *n*-hexane. Wash the first separator with two 30-ml portions of warm distilled water and add these washings to the second separator.

### D 129 – 52,[3] Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method:

*New Note 1.*—Add a new Note 1 immediately after the weight tables in Section 5 (*a*), renumbering the present Note 1 and subsequent Notes, to read as follows:

NOTE 1.—Use of sample weights containing over 20 mg of chlorine may cause corrosion of the bomb. To avoid this, it is recommended that for samples containing over 2 per cent chlorine, the sample weight be based on the chlorine content as given in the following table:

| Chlorine Content, per cent | Weight of Sample, g | Weight of White Oil, g |
|---|---|---|
| 2 to 5 | 0.4 | 0.4 |
| 5 to 10 | 0.2 | 0.6 |
| 10 to 20 | 0.1 | 0.7 |
| 20 to 50 | 0.05 | 0.7 |

*Section 7.*—Add a new Note 5 to the end of Section 7 to read as follows:

NOTE 5.—The precision shown in the above table does not apply to samples containing over 2 per cent chlorine because an added restriction on the amount of sample which can be ignited is imposed.

### D 288 – 53,[3] Definitions of Terms Relating to Petroleum:

*New Definition.* Add the following new definition:

*Extraction Naphtha.*—A naphtha suitable for extraction purposes.

NOTE.—Specifications for extraction naphtha appear in Method D 855, Analysis of Oil-Soluble Sodium Petroleum Sulfonates, and Method D 1216, Analysis of Calcium and Barium Petroleum Sulfonates.

### D 666 – 53,[3] Conversion of Kinematic Viscosity to Saybolt Furol Viscosity:

The method has been rewritten to include a conversion to Saybolt Furol viscosity at 210 F and is appended hereto.[2] A supplement describing the coöperative work upon which the revision is based is also appended hereto.[1]

### D 1094 – 53,[3] Test for Water Tolerance of Aircraft Fuels:

*Title.*—Change to read: "Water Tolerance of Aviation Fuels."

*Section 1.*—Change "jet turbine" to "aviation turbine engine."

*Section 3.*—Change to read as follows:

3. Measure 20 ml of distilled water at room

temperature in a 100-ml glass-stoppered graduated cylinder, and record the volume to the nearest 0.5 ml. The distilled water may be buffered with a phosphate solution to a pH of approximately 7. In the event a phosphate buffer is used, the final buffered solution shall contain 1.15 g of potassium monohydrogen phosphate ($K_2HPO_4$) and 0.46 g of potassium dihydrogen phosphate ($KH_2PO_4$) per 100 ml of distilled water. Add 80 ml of the fuel to be tested, shake vigorously for at least 2 min, and allow to settle undisturbed on a vibration-free surface for a maximum period of 5 min. Caution should be exercised to avoid swirling during or at the end of the agitation period because the swirling action tends to break any emulsion that might be formed.

## TENTATIVES CONTINUED WITHOUT REVISION

The committee has reviewed the following 14 tentatives which have not been revised for at least one year and, for good and sufficient reason discussed within the committee, recommends that they be continued as tentative without revision:

**D 285 - 54 T,** Test for Distillation of Crude Petroleum,
**D 445 - 53 T,** Test for Kinematic Viscosity,
**D 483 - 52 T,** Test for Unsulfonated Residue of Petroleum Plant Spray Oils,
**D 611 - 55 T,** Test for Aniline Point and mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents,
**D 613 - 56 T,** Test for Ignition Quality of Diesel Fuels by the Cetane Method,
**D 614 - 56 T,** Test for Knock Characteristics of Aviation Fuels by the Aviation Method,
**D 875 - 53 T,** Calculation for Olefins and Aromatics in Gasoline,
**D 909 - 49 T,** Test for Knock Characteristics of Aviation Fuels by the Supercharge Method,
**D 975 - 53 T,** Test for Classification of Diesel Fuel Oils,
**D 1018 - 55 T,** Test for Hydrogen in Petroleum Fractions by the Lamp Method,
**D 1022 - 49 T,** Test for Peroxides in Butadiene (Ferrous-Titanous Method),
**D 1263 - 53 T,** Test for Leakage Tendencies of Automotive Wheel Bearing Greases,
**D 1264 - 53 T,** Test for Water Washout Characteristics of Lubricating Greases, and
**D 1368 - 55 T,** Test for Trace Concentrations of Tetraethyllead in Primary Reference Fuels.

## AMERICAN STANDARDS

The committee recommends the following for consideration by ASA Sectional Committee Z-11 on Petroleum Products and Lubricants:

*Approval as American Standard:*

Test for Analysis of 60 Octane Number *Iso*-octane-Normal Heptane ASTM Knock Test Reference Fuel Blends by Infrared Spectrophotometry (D 1095 - 54),
Test for 1,3-Butadiene in $C_4$ Hydrocarbon Mixtures by Ultraviolet Spectrophotometry (D 1096 - 54),
Test for Density and Specific Gravity of Liquids by Bingham Pycnometer (D 1217 - 54), and
Test for Unsaturated Light Hydrocarbons (Silver-Mercuric Nitrate Method) (D 1268 - 55).

*Reapproval as American Standard as Revised in This Report:*

Test for Melting Point of Paraffin Wax (D 87 - 42; Z11.4—1942),
Test for Cloud and Pour Points (D 97 -47; Z11.5—1948),
Test for Flash and Fire Points by Cleveland Open Cup (D 92 - 56; Z11.6—1956),
Test for Heat of Combustion of Liquids by Bomb Calorimeter (D 240 - 50; Z11.14—1950),
Definitions of Terms Relating to Petroleum (D 288 - 53; Z11.28—1953),
Test for Existent Gum in Fuels by Jet Evaporation (D 381 - 56 T; Z11.36—1953),
Test for Distillation of Plant Spray Oils (D 447 55 T; Z11.43—1955),
Conversion of Kinematic Viscosity to Saybolt Furol Viscosity (D 666 - 53; Z11.53—1953),
Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method) (D 873 - 49; Z11.60—1949),
Test for Sulfated Residue from New Lubricating Oils (D 874 - 55; Z11.68 - 1955)
Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (D 1019 - 56 T; Z11.71—1956), and
Test for Water Tolerance of Aircraft Fuels (D 1094 - 53; Z11.82—1953).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The report has been submitted to letter ballot of the committee, which consists of 128 voting members; 99 members returned their ballots, of whom 92 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. M. SMITH,
*Chairman.*

W. T. GUNN,
*Secretary.*

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards revisions of the Tentative Specifications for Gasoline (D 439 - 56 T). The revisions were accepted by the Standards Committee on August 26, 1957, and the revised specifications appear in the 1957 Supplement to Book of ASTM Standards, Part 5, and in the Compilation of ASTM Standards on Petroleum Products and Lubricants.

# REPORT OF COMMITTEE D-4
## ON
# ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials held two meetings during the year: in Atlantic City, N. J., on June 20, 1956, and in Philadelphia, Pa., on February 8, 1957.

The committee has unanimously elected E. F. Kelley as an Honorary Member of the committee.

At the 1956 Annual Meeting, Committee D-2 on Petroleum Products and Lubricants presented to the Society the recommendation for the revision and reversion to tentative of Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 – 46). This method is also under the jurisdiction of Committee D-4, and while no similar recommendation had been presented to the Society at that time by Committee D-4, these revisions now have been approved by Committee D-4.

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Tentative Method of Test for:*

Moisture or Volatile Distillates in Bituminous Mixtures (D 1461 – 57 T), and

*Revision and Reversion to Tentative of:*

Standard Specifications for Calcium Chloride (D 98 – 48).

The recommendation with respect to Standard Specifications D 98 – 48 was accepted by the Administrative Committee on November 15, 1956, and the revised specifications appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

The new Tentative Method D 1461[1] was accepted by the Administrative Committee on May 15, 1957.

### REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

**Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 42 T):**[2] Revise as appended hereto.[1]

These methods are under joint jurisdiction with Committee D-18 on Soils for Engineering Purposes, and the proposed revisions were presented to the Society by Committee D-18 at the 1956 Annual Meeting. However, since the revisions had not been acted upon by Committee D-4 at that time, they were withheld from publication and are now being submitted to the Society by Committee D-4.

**Tentative Specifications for Crushed Stone, Crushed Slag, and Gravel for Single or Multiple Bituminous Surface Treatment (D 1139 – 55 T):**[2] Revise as appended hereto.[1]

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new and revised tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

## REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Specifications for Crushed Stone, Crushed Slag, and Gravel for Water-Bound Macadam Base and Surface Courses of Pavements (**D 694 - 55**):[2] Revise as appended hereto.[1]

Standard Specifications for Mineral Filler for Sheet Asphalt and Bituminous Concrete Pavements (**D 242 - 39**):[2] Revise as appended hereto.[1]

Standard Method of Test for Soft Particles in Coarse Aggregates (**C 235 - 54**):[2] Revise as appended hereto.[1] (Jointly with Committee C-9 on Concrete and Concrete Aggregates.)

## ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Bitumen Content of Paving Mixtures by Centrifuge (**D 1097 - 54 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without change.

## ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions issued June, 1956,[3] of the Standard Specifications for Emulsified Asphalt (**D 977 - 53**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

## REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Cement Content of Soil-Cement Mixtures (**D 806 - 47**)[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society. (This recommendation is made jointly with Committee D-18 on Soils for Engineering Purposes.)

*Section 5.*—Replace Paragraphs (*g*) and (*h*) with the following new Paragraph (*g*), and reletter the subsequent paragraphs accordingly:

(*g*) Wash the precipitate back into the original beaker, add 200 ml of water and 10 ml of $H_2SO_4$ (1:1). Heat the solution just short of boiling, and titrate it with the standard $KMnO_4$ solution (Note 1) to a permanent pink color. Add the filter paper and macerate it. Continue the titration slowly until the pink color is permanent to 10 sec.

NOTE 1.—The temperature of the standard $KMnO_4$ solution should not vary from its standardization temperature so much as to cause a serious error in the determination of CaO. At ordinary room temperatures the volume of pure water changes to the extent of 0.01 to 0.04 per cent for each degree Centigrade, depending on the temperature.

Reletter Paragraph (*j*) as Paragraph (*i*), and change to read as follows:

(*i*) *Alternate Titration Procedure.*—Titrate as described in Paragraph (*g*), except that the $KMnO_4$ solution need not be a standard 0.1 *N* solution but the same solution shall be used in titrating all the components. Omit the blank determination described in Paragraph (*h*).

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentative specifications and methods:

*Tentative Methods of:*

Test for Loss on Heating of Oil and Asphaltic Compounds (**D 6 - 39 T**),

Testing Soil-Bituminous Mixtures (**D 915 - 47 T**),

Test for Shear Strength of Flexible Road Surfaces, Subgrades and Fills by the Burggraf Shear Apparatus (**D 916 - 47 T**),

Test for Vacuum Distillation of Liquid and Semi-Solid Asphaltic Materials to Obtain a Residue of Specified Penetration (**D 1189 - 52 T**),

Testing Concrete Joint Sealers (**D 1191 - 52 T**),

*Tentative Specifications for:*

Asphalt Cement for Use in Pavement Construction (**D 946 - 47 T**), and

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[3] 1956 Supplement to Book of ASTM Standards, Part 3.

Concrete Joint Sealer, Hot-Poured Elastic Type (**D 1190 – 52 T**).

### EDITORIAL CHANGE

The committee recommends editorial changes as follows in the Standard Method of Test for Sieve Analysis of Mineral Filler (**D 546 – 55**):[2]

*Section 2 (b)*.—Delete the note to this section.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee A-4 on Papers* (W. H. Goetz, chairman) was established during the year and has arranged for presentation at the 1957 Annual Meeting of the Society a paper by C. E. Proudley and H. Fred Waller, Jr., entitled "A Cooperative Investigation of the Method of Test for Compressive Strength of Bituminous Mixtures."

*Subcommittee B-2 on Physical Tests for Compressed Bituminous Mixtures* (L. F. Rader, chairman) has drafted for study methods of test for stability and flow measurements by the Marshall Test apparatus, stabilometer and cohesiometer test measurements by the Hveem apparatus, and a method of compaction of bituminous specimens by the kneading compactor.

The subcommittee considered a suggested modified method for determination of specific gravity of compressed bituminous mixtures (rubber bag method), but in the future all matters pertaining to specific gravity measurements will be under the jurisdiction of Subcommittee B-14.

*Subcommittee B-3 on Distillation* (J. W. Donegan, chairman) completed its work on proposed revisions of the Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 – 46) referred to earlier in the report. It is continuing work on a statement on precision for the Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 – 55). A comparison is being made of Method D 402 and the British Method IP 27/56 looking toward having these two procedures technically equivalent.

*Subcommittee B-5 on Softening Point* (D. F. Fink, chairman) is revising the Method of Test for Softening Point of Bituminous Materials (D 36 – 26). A cooperative testing program has been carried out in eight laboratories using the method contained in the proposed tentative revision, and the results are being studied.

*Subcommittee B-6 on Extraction and Recovery of Constituents from Bituminous Mixtures* (E. W. Klinger, chairman) has recommended the adoption as standard of Tentative Method of Test for Bitumen Content of Paving Mixtures by Centrifuge (D 1097 – 54 T). It plans to continue investigations on methods of extraction by centrifuge, reflux, immersion, and immersion with reflux.

*Subcommittee B-7 on Viscosity and Float Test* (C. A. Benning, chairman) is developing a method for the determination of specific viscosity of tars and tar products by use of the Engler viscosimeter. A study of cooperative work done several years ago has indicated a way of simplifying the method and a new draft has been prepared.

*Subcommittee B-9 on Size of Aggregates* (Fred Hubbard, chairman) prepared the editorial change in the Method of Test for Sieve Analysis for Mineral Filler (D 546 – 55) referred to earlier in the report.

*Subcommittee B-12 on Structural Properties of Mineral Aggregates* (Stanton Walker, chairman) prepared the revisions in the Method of Test for Soft Particles in Coarse Aggregates (C 235 – 54) referred to earlier in the report.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

## TABLE I.—GRAND AVERAGE OF VALUES FOR PROPERTIES IN THIN FILM TESTS.

| Sample | Tests on Original Material | | | Thin Film Tests | | | |
|---|---|---|---|---|---|---|---|
| | | | | | Tests on Residue | | |
| | Penetration at 77 F | Softening Point, deg Fahr | Ductility at 39.2 F and 1 cm per min, cm | Loss on Heating, per cent | Penetration at 77 F | Softening Point, deg Fahr | Ductility at 39.2 F and 1 cm per min, cm |
| A | 90.5 | 113.2 | ... | 0.127 | 60.3 | 121.1 | 10.6 |
| B | 93.5 | 117.0 | 22.2 | 1.24 | 51.1 | 131.6 | 4.8 |
| C | 80.2 | 121.0 | 6.1 | +0.050[a] | 51.8 | 137.6 | 3.7 |
| D | 84.9 | 115.9 | 14.8 | +0.052[a] | 57.8 | 124.4 | 5.7 |

[a] This figure was actually a gain in weight rather than a loss.

## TABLE II.—STATISTICAL ANALYSIS OF THIN-FILM OVEN TEST DATA.

| Sample | For Repeatability | | | | For Reproducibility | | | |
|---|---|---|---|---|---|---|---|---|
| | Penetration at 77 F | Softening Point, deg Fahr | Ductility at 39.2 F and 1 cm per min, cm | Loss on Heating, per cent | Penetration at 77 F | Softening Point, deg Fahr | Ductility at 39.2 F and 1 cm per min, cm | Loss on Heating, per cent |
| **TEST PROPERTIES OF ORIGINAL MATERIAL—STANDARD DEVIATION FROM THE MEAN** | | | | | | | | |
| 90834[a] | ... | ... | ... | ... | 3.1 | 3.1 | ... | ... |
| 91911[a] | ... | ... | ... | ... | 8.4 | 1.6 | ... | ... |
| A | 1.3 | 0.7 | [b] | ... | 3.6 | 2.1 | [b] | ... |
| B | 0.8 | 1.9 | 1.4 | ... | 2.5 | 1.7 | 10.6 | ... |
| C | 1.2 | 1.3 | 0.5 | ... | 3.7 | 1.7 | 0.8 | ... |
| D | 1.1 | 1.2 | 2.4 | ... | 2.8 | 1.7 | 5.7 | ... |
| Average | 1.1 | 1.3 | 1.4 | ... | 4.0 | 2.0 | 5.7 | ... |
| **TEST PROPERTIES FOR THIN FILMS—STANDARD DEVIATION FROM THE MEAN** | | | | | | | | |
| 90834[a] | 1.9 | 1.1 | ... | 0.049 | 3.7 | 2.0 | ... | 0.080 |
| 91911[a] | 2.6 | 0.9 | ... | 0.25 | 2.4 | 2.3 | ... | 0.37 |
| A | 1.1 | 1.3 | 1.7 | 0.04 | 2.4 | 1.5 | 3.1 | 0.04 |
| B | 2.2 | 1.8 | 0.8 | 0.07 | 3.4 | 2.7 | 1.3 | 0.22 |
| C | 1.8 | 1.5 | 0.4 | 0.016 | 3.2 | 1.7 | 0.3 | 0.017 |
| D | 2.4 | 1.4 | 0.5 | 0.017 | 3.3 | 1.9 | 1.0 | 0.018 |
| Average | 2.0 | 1.3 | 0.9 | 0.07 | 3.1 | 2.0 | 1.4 | 0.12 |

[a] Data from first series.

[b] Data not suitable for analysis.

## TABLE III.—REPRODUCIBILITY OF THE PERCENTAGE RETAINED PENETRATIONS FOR THE THIN FILM TEST.[a]

| Sample | Standard Deviation from the Mean for Reproducibility, percentage Retained Penetration | 95% Confidence Limits, percentage retained penetration |
|---|---|---|
| A | 2.6 | 8.3 |
| B | 2.5 | 8.0 |
| C | 3.6 | 11.8 |
| D | 4.5 | 14.1 |
| | | Average 10.6 |

[a] The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount: Reproducibility = $\frac{\text{percentage retained penetration}}{10}$

*Subcommittee B-14 on Specific Gravity of Fine and Coarse Aggregates* (H. T. Williams, chairman) is initiating work on specific gravity of aggregates, bituminous mixtures, and bituminous materials.

*Subcommittee B-16 on Setting Qualities of Bituminous Materials* (A. B. Cornthwaite, chairman) is continuing its study of the variables in a proposed rolling ball test for evaluating the setting qualities of cut-back asphalts. Test results from a cooperative study by five laboratories indicated repeatability of the method was very good but better reproducibility between laboratories is desired.

*Subcommittee B-17 on Emulsion Tests* (R. R. Thurston, chairman) is preparing repeatability and reproducibility sections for addition to Method of Testing Emulsified Asphalts (D 244 – 55). A new method of stone coating and water stripping tests for use with MS emulsions is being studied.

*Subcommittee B-19 on Accelerated Tests for Durability of Bituminous Materials* (D. C. Taylor, chairman) has prepared for publication as information only a Proposed Method for determining the effect of heat and air on asphaltic materials when exposed in thin films.[5] The results of tests carried out to determine its reproducibility and repeatability are given in Tables I, II, and III. A task group is carrying out cooperative testing with the Sliding Plate Microviscometer developed by the Shell Development Co.

*Subcommittee B-24 on Penetration Test* (J. Y. Wellborn, chairman) is working to improve the precision of the Penetration Test (D 5 – 52) adding provisions in the test procedure, and redesigning the penetration needle to provide better control.

*Subcommittee B-26 on Effect of Water on Bituminous Coated Aggregates* (C. M. Hewett, chairman) is investigating stripping test procedures. Work is also being continued on the investigation of other methods such as the lithium tracer-flame photometer procedure.

*Subcommittee C-4 on Emulsified Asphalts* (C. E. Proudley, chairman) recommended the adoption as standard of tentative revisions of the Specifications for Emulsified Asphalt (D 977 – 53) referred to earlier in the report.

*Subcommittee C-8 on Water-Bound Roads* (R. R. Litehiser, chairman) recommended the revision and reversion to tentative of Standard Specifications for Crushed Stone, Crushed Slag, and Gravel for Water-Bound Macadam Base and Surface Courses for Pavements (D 694 – 55) referred to earlier in the report. The principal revisions limit the proposed specifications to base courses, including dry-bound macadam.

*Subcommittee C-10 on Plant-Mix Bituminous Surfaces and Bases* (Fred Hubbard, chairman) has prepared extensive revisions of the Specifications for Mineral Filler for Sheet Asphalt and Bituminous Concrete Pavements (D 242 – 39) which are referred to earlier in the report. The subcommittee is continuing a detailed study of the Specifications for Hot-Mixed, Hot-Laid Asphaltic Concrete for Base and Surface Courses (D 947 – 55).

*Subcommittee C-12 on Bituminous Surface Treatments* (J. O. Izatt, chairman) has proposed a number of revisions of the Specifications for Crushed Stone, Crushed Slag, and Gravel for Single and Multiple Bituminous Surface Treatments (D 1139 – 55 T) which are referred to earlier in the report. The revisions describe deleterious substances in terms of ASTM test methods now employed for the control of other aggregates and establishes limits for permissible amounts of each type.

*Subcommittee C-13 on Bituminous*

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[5] See p. 328.

*Paving Plant Operations* (I. H. Boggs, chairman) is working on modifications of the Specifications for Bituminous Mixing Plant Requirements (D 995 - 55) to provide for better screening efficiency in bituminous mixing plants.

*Subcommittee D-1 on Calcium Chloride and Sodium Chloride for Use As Road Materials* (H. F. Clemmer, chairman) is preparing new specifications for sodium chloride, which have been submitted to members of Committee D-4 as information only.

*Subcommittee D-2 on Highway Traffic Markers (Paints, Insets and Strips)* (N. G. Smith, chairman) is developing a use-performance type of specification for traffic marking paints. It has temporarily discontinued work on permanent type traffic markers.

*Subcommittee D-3 on Expansion Joint Materials* (H. F. Clemmer, chairman) is revising the Specifications for Preformed Expansion Joint Fillers for Concrete (Non-extruding and Resilient Types) (D 544 - 56 T) to incorporate a specification on jet fuel resistant joint sealers and revisions of the method of test for binding media of cork expansion joint.

*Joint Subcommittee of Committees D-4 and D-18 on Tests and Specifications for Stabilized Soils* (H. F. Clemmer, chairman) prepared revisions of the Method of Test for Moisture-Density Relations of Soils (D 698 - 42 T) referred to earlier in the report, and it has proposed changes in the Method of Test for Cement Content of Soil-Cement Mixtures (D 806 - 47).

This report has been submitted to letter ballot of the committee, which consists of 165 members; 112 members returned their ballots, of whom 111 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

K. B. WOODS,
*Chairman.*

J. M. GRIFFITH,
*Secretary.*

# APPENDIX

## PROPOSED METHOD OF TEST FOR DETERMINING EFFECT OF HEAT AND AIR ON ASPHALTIC MATERIALS WHEN EXPOSED IN THIN FILMS[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is intended for the determination of the effect of heat and air on asphaltic materials when heated in thin films as prescribed in the method. The loss in weight and a comparison of the penetration, softening point, and ductility before and after heating are used as a measure of the effect of this test on asphaltic materials.

NOTE. 1.—For additional information of the changes that occur in the asphaltic materials, solubility and Oliensis spot tests also may be used.

NOTE 2.—While this test is prescribed primarily for asphalt cements, it may also be useful for cut-back asphalts. For the latter material, the test should be made on the residue from distillation obtained by the Method of Test for Distillation of Cut-Back Asphaltic Products (ASTM Designation: D 402).[3]

### Apparatus

2. (*a*) *Oven.*—An oven conforming to the oven specified in the Method of Test for Loss on Heating of Oil and Asphaltic Compounds (ASTM Designation: D 6)[3] with the following exception: The circular shelf shall be constructed so that it will support one or more sample containers, 5.5 in. in diameter, in a horizontal position.

(*b*) *Thermometer.*—An ASTM Loss on Heat Thermometer having a range of 155 to 170 C and conforming to the requirements for thermometer 13C, as prescribed in ASTM Specifications E 1.[3]

(*c*) *Container.*—A cylindrical pan, 5.5 in. in inside diameter and $\frac{3}{8}$ in. deep with a flat bottom, made of aluminum 0.0299 in. thick or of stainless steel not lighter than 26 USS gage. Fifty milliliters of the sample in this size container gives a film thickness of approximately $\frac{1}{8}$ in.

### Procedure

3. (*a*) Weigh 50.0 ± 0.5 ml of the bituminous material, the weight of which shall be calculated from the specific gravity of the material, into a tared 5.5-in. aluminum pan (Note 3). Semisolid materials should be heated to a fluid condition previously, at a temperature not more than 100 F above the softening point. If the material has been heated to facilitate transfer, cool the container with the sample to room temperature and weigh to the nearest 0.001 g. With the oven at 325 F (163 C), place the container with the sample on the circular shelf, close the oven,

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[1] This proposed method is under the jurisdiction of the ASTM Committee D-4 on Road and Paving Materials.

[2] Published as information, June, 1957.

[3] 1955 Book of ASTM Standards, Part 3.

and rotate the shelf at a rate of 5 to 6 rpm. The temperature shall be maintained at 325 ± 1.8 F (163 ± 1 C) for 5 hr after the sample has been introduced and the oven has again reached that temperature. The 5-hr period shall start when the temperature reaches 324 F (162 C), and in no case shall the total time that a sample is in the oven be more than 5 hr and 15 min. At the conclusion of the heating period, remove the sample from the oven, cool to room temperature, weigh to the nearest 0.001 g, and calculate the loss due to heating.

NOTE 3.—In order to provide sufficient material for tests on the residue, it is often necessary to make the thin-film oven test in duplicate.

(*b*) Determine the temperature of oven by means of the thermometer supported from the shaft of the circular shelf in a vertical position at a point equidistant from the center and the outer edge of the shelf.

(*c*) After weighing the sample, place it on the circular shelf of the oven together with the 3- or 6-oz ointment can resting on a piece of asbestos cement industrial board (Transite). Bring the oven to a temperature of 325 F (163 C) prior to placing the sample, can, and Transite plate on the shelf. Close the oven and rotate the shelf for 15 min. Remove the sample, can, and Transite plate, and immediately pour the sample into the ointment can. Remove all material from the 5.5-in. container by scraping with a putty knife. If duplicate tests are made, combine the residues. Stir the sample thoroughly and test by the following methods:

*Penetration.*—Method of Test for Penetration of Bituminous Materials (ASTM Designation: D 5).[3]

*Softening Point.*—Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (ASTM Designation: D 36).[3]

*Ductility.*—Method of Test for Ductility of Bituminous Materials (ASTM Designation: D 113).[3]

NOTE 4.—If it is necessary to reheat the residue, this should be done at the lowest possible temperature.

## Precision

4. The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

$$\text{Reproducibity} = \frac{\text{percentage of retained penetration}}{10}$$

# REPORT OF COMMITTEE D-5
# ON
# COAL AND COKE*

Committee D-5 on Coal and Coke held one meeting during the year: in Atlantic City, N. J., on June 21, 1956; in addition, a meeting of the Advisory Subcommittee was held in Urbana, Ill., on September 4, 1956, and subcommittee meetings were held in Philadelphia, Pa., on February 4 to 8, 1957.

The committee lost two members, one by retirement and the other by death. Five members were added making a total voting membership of 62, of whom 15 are classified as producers, 24 as consumers, and 23 as general interest members.

Committee D-5, through the American Standards Association, has continued active participation in the work of Technical Committee 27 on Solid Mineral Fuels of the International Organization for Standardization. No meetings of this ISO Committee have been held during the past year, but many documents have been received and studied. Opinions representative of the thinking of U.S.A. technologists have been submitted to the Secretariat for consideration. A meeting of Working Group 7 on Sampling was held in London, England, on November 26 to 28, 1956, at which the United States was represented by Bertrand A. Landry, Manager of Battelle Memorial Inst. in Paris, France.

## ADOPTION OF TENTATIVE REVISION AS STANDARD

Committee D-5 recommends that the tentative revisions issued in June, 1954,[1] of the Standard Method of Test for Free Swelling Index of Coal (**D 720 – 46**) be approved for reference to letter ballot of the Society for adoption as standard. This revision is related to the measurement of the swelling index number of buttons, the shapes of which do not conform to the standard profiles.

## TENTATIVE REVISION OF STANDARD

Committee D-5 recommends tentative revisions as follows of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (**D 271 – 48**):[1]

*Section 39.*—There being no satisfactory direct ASTM method of determining oxygen, it is calculated by subtracting from 100 the sum of the percentages of hydrogen, carbon, nitrogen, sulfur, moisture, and ash. The result so obtained is affected by the errors incurred in the other determinations and also by the changes in weight of the ash-forming constituents on ignition.

## TENTATIVES CONTINUED WITHOUT REVISION

Committee D-5 recommends the continuation without revision of all other tentatives and tentative revisions of standards under its jurisdiction.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee II on Nomenclature and*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 5.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Definitions* (C. H. Sawyer, chairman) is reviewing terms selected from current standards and tentatives under the jurisdiction of the Committee D-5 about which some question has been raised as to their adequacy of meaning.

*Subcommittee VII on Pulverizing Characteristics of Coal* (H. F. Yancey, chairman) is considering revision of the Standard Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 51) to include a standard grindability sample for calibration of the equipment.

*Subcommittee XV on Plasticity and Swelling of Coal* (Michael Perch, chairman) recommended the adoption as standard of the tentative revision of Standard Method of Test for Free Swelling Index of Coal (D 720 - 46) referred to earlier in the report. Methods of electrical heating for this test are being investigated.

Repeatability and reproducibility data for the Audibert-Arnu dilatometer test were received from ISO/TC 27 for study.

Research work on the effect of certain variables on the Gieseler Plastometer Test was completed and a third draft of the test is in preparation. Further work is planned to improve the operation of the plastometer.

*Subcommittee XX on Sampling and Fineness Test of Pulverized Coal* (E. L. Rogers, chairman) has continued its work on the revision of ASTM Method of Sampling and Fineness Test of Powdered Coal (D 197 - 30). Further data are necessary before reaching final conclusions and arrangements have been made to secure these data. The subcommittee is also considering a proposed change in title of this method to include sampling and fineness testing of solid fuels other than coal.

*Subcommittee XXI on Methods of Analysis* (R. F. Abernethy, chairman) prepared the proposed tentative revision of Standard Methods D 271 - 48 referred to earlier in the report.

The subcommittee is revising the method appearing in D 271 for determining fusibility of ash to include an oxidizing atmosphere. New methods are being developed for forms of sulfur in coal, chlorine in coal, sulfur in coal ash, and mineral carbon dioxide in coal.

*Subcommittee XXIII on Sampling* (W. M. Bertholf, chairman).—One of the largest projects now under way in Committee D-5 is the study of mechanical methods for sampling large tonnages of coal. The study includes collecting information on methods of statistical analysis of experiments to determine the precision of the process of preparing and analyzing coal or coke samples. Data on the relationship between increment weights and variance for coal and on the reduction and analysis of coal samples are also being assembled and evaluated.

This report has been submitted to letter ballot of the committee, which consists of 62 voting members; 59 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

O. W. REES,
*Chairman.*

C. C. RUSSELL,
*Secretary.*

# REPORT OF COMMITTEE D-6
## ON
# PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 20, 1956, in Atlantic City, N. J., and on February 21, 1957, in New York, N. Y.

The Joint TAPPI-ASTM Committee on Coordination, under the chairmanship of R. H. Carter, met at the Lorraine Hotel, Madison, Wis., on September 11, 1956, to discuss the coordination activities of the two Societies. The governing bodies of the two groups have approved the objectives of the joint committee.

The committee will sponsor a symposium on "New Developments in Paper and Paper Testing" to be presented in June, 1958. Henry A. Birdsal is in charge of the arrangements.

The committee expresses its gratitude and appreciation to Everett G. Ham for his leadership and guidance these many years. Mr. Ham has retired.

The committee consists of 93 members, of whom 77 are voting members; 33 are classified as producers, 23 as consumers, and 37 as general interest members.

### REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Reducible Sulfur in Paper (**D 984 - 50**),[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

*Section 3(c)*.—Change to read as follows:

(*c*) *Absorbent Cotton.*—Surgical grade from a freshly opened package which has not been exposed to laboratory fumes. Handle with forceps only.

*Section 6*.—In Paragraph (*a*), the thirteenth and twenty-first lines, delete the word "prepared." In Paragraph (*b*), the fourth line, change "sulfur-free" to read "absorbent."

### TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

*Tentative Method of Test for:*

Water Resistance of Paper, Paperboard and Other Sheet Materials by the Dry-Indicator Method (**D 779 - 55 T**),
Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (D 781 - 44 T),
Stretch of Paper and Paper Products Under Tension (**D 987 - 48 T**),
Water Vapor Permeability of Paper and Paperboard (**D 988 - 51 T**),
Chloride Content of Paper Products (D 1161 - 51 T),
Pinholes in Glassine and Other Grease Proof Papers (**D 1221 - 52 T**),
Contrast Gloss of Paper at 57.5 deg (**D 1222 - 52 T**),
Specular Gloss of Paper at 75 deg (**D 1223 - 52 T**), and
Dimensional Changes of Paper with Changes in Moisture Conditions (**D 1270 - 53 T**).

### EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentatives:

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 7.

### Standard Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 48):[1]

*New Section.*—Add a new Section 2 on Significance of Test as follows and renumber the present Section 2 and subsequent sections accordingly:

2. This method identifies the mineral constituents of a sheet of paper used in the body of the web (commonly known as "fillers") or in the coating applied to the web. While the analytical procedures actually serve to identify only the ions of such minerals, the results can be interpreted in terms of the minerals themselves.

### Standard Method for Quantitative Determination of Coating on Mineral-Coated Paper (D 687 – 44):[1]

*New Section.*—Add a new Section 2 on Significance of Test and renumber the present Section 2 and subsequent sections accordingly:

2. *Significance of Test.*—The weight of coating applied to a raw stock may have a marked effect on the strength and printability of the coated sheet, as well as being a factor in various optical properties such as opacity, brightness and gloss. Therefore the significance of coating weight as a factor in control, acceptance and research is apparent.

*Section 6(a).*—Delete the two examples given in parentheses.

### Standard Method of Test for Titanium Dioxide in Paper (D 921 – 49):[1]

*New Section.*—Add a new Section 2 on Significance of Test and renumber the present Section 2 and subsequent sections accordingly:

2. *Significance of Test.*—Since titanium dioxide is one of the most expensive mineral constituents used in paper, it is frequently desirable to know how much is present. This is particularly important when the pigment is used as a beater filler where there is always some question as to how much is actually being retained in the web. The method therefore has significance in control, in research where the effect of various degrees of pigmentation is to be determined, and in acceptance since papers in which titanium dioxide is a component usually sell at a premium price.

### Tentative Method of Test for Resistance of Paper to Passage of Air (D 726 – 55 T):[1]

*New Section.*—Revise Section 2 on Significance as follows:

2. The air resistance of paper can be used as an indirect indication of such variables as degree of beating, absorbency (penetration of oil, water, etc.), apparent specific gravity, filtering efficiency for liquids or gases, etc. It is influenced by both the internal structure and the surface finish. Internal structure is controlled largely by type and length of fiber, degree of hydration, orientation, and compaction. Surface finish is governed by the degree of wet pressing, types of felts used in manufacture and, primarily, calendering, as well as by the presence or absence of coating. Air resistance is further greatly influenced by the type and amount of filler, sizing, etc. The air resistance of coated papers is generally so high as to be of doubtful significance. While the measurement of air resistance is a useful control test for machine production, due to the number and complexity of factors outlined above, careful judgment with a consideration for end-product use should govern the adoption of specification limits for air resistance.

In insulating papers, air resistance can be used as a criterion in predicting dielectric strength and absorbency; in uncoated printing papers it can be used as an indication of rotogravure ink absorption; in filter papers it can foretell the performance in filtering liquids or gases.

### Standard Method of Test for Chloride Content of Paper and Paper Products (D 1161 – 51 T):[1]

*Section 1.*—Add a Note to read as follows:

NOTE.—This method is thought to have certain faults which until resolved make it undesirable for publication as a Standard Method. There is an indication of loss of chlorides in ashing and such loss may be significant at low chloride content (approximately 100 ppm and less). However, it does have some usefulness and is of sufficient value to be retained as a Tentative Method until such time as the necessary work can be done on it to resolve some of the objections, or until a better method is available.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Sampling and Conditioning* (T. W. Lashof, chairman) has organized task groups on sampling, on conditioning rooms and on temperature and humidity effects. They will determine how well standard conditions as defined in Standard Method of Conditioning Paper and Paper Products for Testing (D 685 - 44) are being maintained in existing conditioning rooms, and will study the effect of humidity and temperature on the physical properties of paper to determine whether the existing conditioning room requirements are appropriate.

*Subcommittee II on Chemical Test Methods* (A. P. Tracy, chairman) is working on two new test methods, and is improving existing tests for starch in paper, for qualitative examination of mineral filler and mineral coating, for titanium dioxide and for chloride content of paper.

*Subcommittee III on Physical Test Methods for Paper* (L. Price, chairman) is revising the Method of Test for Tensile Breaking Strength of Paper and Paperboard (D 828 - 48). A joint TAPPI-ASTM task group is working on the Method of Test for Basis Weight of Paper and Paperboard (D 646 - 50). An investigation of the effect of different thicknesses of diaphragm plates is directed toward a revision of the Method of Test for Bursting Strength of Paper (D 774 - 46).

*Subcommittee IV on Physical Test Methods for Container Board* (W. B. Lincoln, Jr., chairman).—A task group is reviewing the work of TAPPI, Forest Products Laboratory, the Institute of Paper Chemistry, and others as a guide to future improvement of the Method of Test for Flat Crush of Corrugated Paperboard (D 1225 - 54). The task group reviewing the Method of Test for Puncture and Stiffness of Paperboard, Corrugated and Solid Fiber Board (D 781 - 44 T), will make recommendations for the revision of the drawing of the apparatus, measurement of the stiffness in four directions, a reporting of the tear readings in in.-oz per in. of tear rather than by unit. The study of corrugated medium test and testers will be circulated to the members to elicit comments on a proposed method.

*Subcommittee VI—Editorial* (L. S. Reid, chairman) is continuing the preparation of sections on significance of test for all new and revised methods.

This report has been submitted to letter ballot of the committee, which consists of 78 voting members; 42 members returned their ballots, of whom 37 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. H. CARTER,
*Chairman.*

R. E. GREEN,
*Secretary.*

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE D-7

## ON

## WOOD*

Committee D-7 on Wood met in Philadelphia on February 6 and 7, 1957. The following subcommittee meetings preceded the main committee meeting: Subcommittee I on Specifications for Timber, Subcommittee VIII on Modified Wood and Wood-Base Materials, Subcommittee XIII on Durability and Exposure, Subcommittee XV on Structural Fiberboards; and a meeting of the subcommittee chairmen.

*ISO Proposal on Fiberboards.*—The American Standards Assn. brought to the attention of the ASTM a proposal originated by Norway to form a international committee under the auspices of the Intenational Standardization Organization (ISO) to deal with fiberboards. The proposed scope was as follows: "For the purpose of facilitating international trade, to establish international agreement on standardization of specifications—including terminology, dimensions and tolerances, designations, rules for sampling and methods of tests—for fiber building board." After due consideration of the proposal by Subcommittee XV and by Committee D-7 the committee recommended against its organization with the understanding that the subject could be reviewed again should future developments make it desirable. Substantial results already have been accomplished internationally on fiberboards by the Conference on Wood Technology of the Food and Agriculture Organization of the United Nations, particularly with respect to agreement on methods of test, and attention was also called to the International Consultation on Insulation Board, Hardboard, and Particle Board that was held under the auspices of the FAO at the Palais des Nations, Geneva, Switzerland, January 21–February 5, 1957.

Twelve applicants were elected to membership on the committee; there were seven resignations. Two new consulting members were appointed.

### ASTM STANDARDS FOR APPROVAL AS AMERICAN STANDARDS

The committee recommends that all standards and tentatives under its jurisdiction except the Standard Method of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 – 53) and Tentative Method for Testing Wood Preservatives by Laboratory Soil-Block Cultures (D 1413 - 56 T) be submitted to the American Standards Assn. for approval as American Standard.

### REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

**Tentative Methods for Establishing Structural Grades of Lumber (D 245 - 49 T):**[1] Revise as appended hereto.[2]

**Tentative Specifications for Modified Wood (D 1324 - 54 T):**[1]

*Title.*—Change to read, "Tentative Specifications and Methods of Test for Modified Woods."

*Section 9(a).*—Change to read as follows:

(*a*) The properties of modified wood shall be determined in accordance with the Methods of Testing Veneer, Plywood, and Other Glued Veneer Constructions (ASTM Designation: D 805)[2] or as outlined in Sections 10 to 14 of this specification.

*New Sections.*—Add the following new Sections 10 to 14 under the heading "Hardness Test."

10. *Scope.*—The wide range of densities possible in the manufacture of modified woods makes difficult the selection of a single hardness test method satisfactory for all types of material. The modified Janka ball hardness test described in the Methods of Testing Small Clear Specimens of Timber (ASTM Designation: D 143),[2] has been found satisfactory for the less dense modified woods. The Rockwell hardness test described in the Methods of Testing Veneer, Plywood, and Other Glued Veneer Constructions (ASTM Designation: D 805),[2] may be used for material of medium density. The hardness-penetration test described below has found general acceptance in the industry and can be used successfully for determining the hardness of modified woods varying in density from very light to those of maximum density.

11. *Test Specimen.*—The test specimen shall be a minimum of 2 in. in width and 6 in. in length long, and equal to the thickness of the material.

NOTE.—When the hardness of a thin material is required, it may be necessary to build up the thickness of the test specimen by placing several layers of the same material under the test specimen.

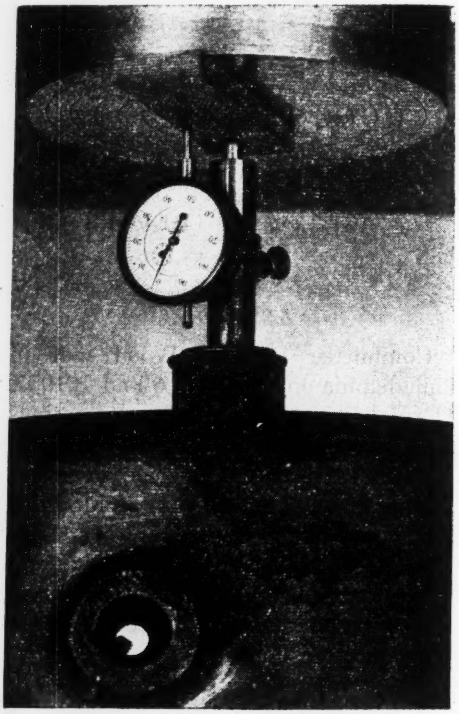

FIG. 1.—Apparatus for Hardness-Penetration Test.

12. *Method of Loading.*—The equipment used for making the hardness-penetration test is shown in Figure 2 (the accompanying Fig. 1). The penetrating tool shall be identical to that specified in ASTM Methods D 143 - 52, except that the collar for determining one-half depth penetration shall be removed. This tool has a 0.444-in. diameter (1 sq cm projected area) penetrating ball. A dial gage, reading to 0.001 in., shall be mounted on the penetrating tool in such a manner that the depth of penetration can be determined. The penetrating tool shall be mounted on a testing machine of suitable capacity and simultaneous readings of load and penetration shall be made at sufficient intervals (at least 10 points or equivalent automatically plotted data), to permit the determination of a straight line relationship. At least 2 such determinations shall be made on each piece. The penetration shall be far enough from the edge to prevent splitting or chipping.

13. *Speed of Testing.*—The load shall be applied to the specimen throughout the test by a uniform motion of the movable head of the testing machine at a rate of 0.02 in. (±25 per cent) per min.

14. *Test Data and Report.*—The hardness of the material shall be reported as the load to cause an arbitrarily selected depth of penetration or as a hardness modulus determined by means of the equation:

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[1] 1955 Book of ASTM Standards. Part 4.

[2] The revised tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 4.

$$H_m = \frac{P}{2\pi rh} = 0.717\frac{P}{h}$$

where:

$H_m$ = load per unit of spherical area of penetration expressed in pounds per square inch,

$r$ = radius of penetration ball (0.222 in.),

$P$ = arbitrarily selected load (in pounds) within linear portion of test data, and

$h$ = depth of penetration in inches at above selected load.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Specifications for Timber* (L. W. Wood, chairman) prepared the revision of the Tentative Methods for Establishing Structural Grades of Lumber (D 245 – 49 T) referred to previously in the report. The subcommittee proposes to continue its studies of stress grading.

*Subcommittee II on Laminated Timber* (Frank J. Hanrahan, chairman) has under way a study to shorten the time required for evaluating the integrity of glue joints. It is anticipated that this will lead to a revision of the Standard Method of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 – 53).

*Subcommittee IV on Wood Paving Blocks* (W. H. O'Brien, chairman) is studying the Specification for Wood Paving Blocks for Exposed Pavements (D 52 – 20), with the objective of revising or discontinuing it because wood blocks are no longer used for exterior paving purposes.

*Subcommittee VII on Wood Poles and Cross Arms* (R. P. A. Johnson, chairman).—The Wood Pole Research Program is the principal activity of Subcommittee VII. Untreated poles in series I and II have been tested, and five interim reports on untreated poles have been distributed by ASTM to the subcommittee members and contributors to the program. Selections of poles for treatment and test in series III have now been completed. All selected poles have been treated except for those of western red-cedar which are now under seasoning preparatory to treatment. Tests of full-size treated southern pine and Douglas-fir poles have been completed. Tests of matched small clear specimen material in southern pine are completed and in Douglas-fir are under way. An interim report on treated southern pine poles is being completed.

Testing of full-size treated poles of western larch and lodgepole pine is going forward, and it is planned to complete all testing by January 1, 1958. The over-all analysis of the data and a final report will then be prepared.

Financial support given the program has been most gratifying. As the last phase of testing proceeds, more than three-quarters of the cost has been underwritten. Further contributions are being solicited, and one new source of substantial support is expected.

Members of Subcommittee VII took part in a Symposium on Wood Poles sponsored by Committee D-7 at the West Coast meeting at Los Angeles in September. Five papers were presented and much interest in the symposium and research program was developed.

The Society is represented on the newly reorganized ASA Sectional Committee 05 on Wood Poles by L. J. Markwardt and W. A. Oliver. Forest Service representatives include R. P. A. Johnson and L. W. Wood, members of Subcommittee VII. An organization meeting of the reactivated Committee 05 was held in November, and plans were made for study and use of data from the ASTM pole project in a complete reconsideration of ASA 05-1948 "Specifications and Dimensions for Wood Poles."

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Further activity in the field of crossarm testing is reported. Crossarm test data and test methods used by Southern California Edison Co. were reviewed and discussed. Plans are being developed for load tests of crossarm constructions at the Forest Products Laboratory in cooperation with the Wisconsin Power and Light Co.

*Subcommittee VIII on Modified Wood and Wood-Base Materials*, (W. G. Youngquist, chairman) prepared the new hardness test referred to previously in the report for addition to the Tentative Specifications for Modified Wood (D 1324 - 54 T). The test method consists of determining simultaneous values of load and penetration of a 0.444-in. diameter steel ball through a specific range of values. A measure of hardness of thin materials, materials of normal density, and high-density materials which would split when subjected to the standard hardness test for wood can be obtained by this new method. The results are reported as a "hardness modulus."

*Subcommittee IX on Moisture Content of Timber* (R. C. Rietz, chairman) is developing more rapid and improved methods of determining the moisture content of treated wood, improved electrical methods for moisture content, and related specifications.

*Subcommittee XIII on Durability and Exposure* (R. M. Lindgren, chairman) has appointed task groups to prepare an accelerated laboratory method of testing the natural decay resistance of woods and a test method for determining the effectiveness of wood preservatives in stakes exposed in field plots.

*Subcommittee XV on Structural Fiberboards* (W. C. Lewis, chairman) expects to complete during the year the preparation of nomenclature and definitions for structural fiber and particle boards.

This report has been submitted to letter ballot of the committee, which consists of 97 voting members: 69 members returned their ballots, of whom 68 have voted affirmatively and 0 negatively.

Respectfully submitted in behalf of the committee,

L. J. MARKWARDT,
*Chairman.*

L. W. SMITH,
*Secretary.*

# REPORT OF COMMITTEE D-8

ON

# BITUMINOUS WATERPROOFING AND ROOFING MATERIALS*

Committee D-8 on Bituminous Waterproofing and Roofing Materials held two meetings during the year: in Atlantic City, N. J., on June 22, 1956, and in Philadelphia, Pa., on February 6, 1957.

The membership, consisting of 79 voting members, is composed of 52 producers, 10 consumers, and 17 general interest members.

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-8 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Methods of Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 - 47) be revised and reverted to tentative status. This recommendation was accepted by the Standards Committee on September 5, 1956, and the revised methods appear in the 1956 Supplement to Book of ASTM Standards, Part 3.

## REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

**Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 - 54 T):**[1] Revise as appended hereto.[2]

**Tentative Specification for Woven Burlap Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1327 - 56 T):**[3]

*Table I.*—Delete "Average loss on heating (exclusive of moisture) of saturated fabric, per cent....4 max."

## TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

**Standard Specifications for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (D 250 - 56):**[3]

*Section 1.*—In the second line, following the word "either," add "with or without perforations."

*Section* 7.—Add to the end of this section, "Perforated Felts shall conform to all requirements of the imperforate type, and in addition shall have uniformly spaced perforations."

*Section* 9.—Change Footnote (*b*) to read as follows:

> [b] Weight of saturant per 100 sq ft shall not be less than 0.40 times the weight of the dry felt for both the 15-lb type and the 30-lb type.

*Section 11.*—Add to the end of this section, "except for intentionally provided perforations and the associated protuberances."

**Standard Specifications for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (D 655 - 47):**[1]

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 3.

[2] The revised tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

[3] 1957 Supplement to Book of ASTM Standards, Part 3.

*Section 9.*—Change Footnote (*a*) to read as follows:

> [a] Weight of saturant per 100 sq ft shall not be less than 0.40 times the weight of the dry felt for both the 20-lb type and the 50-lb type.

### Standard Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 – 47):[1]

*Section 1.*—Change as recommended above for Specifications D 250.

*Section 7.*—Change as recommended above for Specifications D 250.

*Section 11.*—Change as recommended above for Specifications D 250.

### Standard Specifications for Woven Cotton Fabrics Saturated With Bituminous Substances for Use in Waterproofing (D 173 – 44):[1]

*Table 1.*—Delete "Average loss on heating (exclusive of moisture) of asphalt-saturated fabrics ... 4 per cent max."

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives and tentative revisions of the following standards:

*Tentative Specification for:*

Asphalt Insulating Siding Surfaced With Mineral Granules (**D 1226 – 52 T**),
Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (**D 1227 – 52 T**),

*Tentative Method of:*

Testing Asphalt Insulating Siding Surfaced With Mineral Granules (**D 1228 – 52 T**),
Testing Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (**D 1167 – 51 T**),
Test for Staining Properties of Asphalts (Modified Pressure Method) (**D 1328 – 54 T**), and

*Tentative Recommended Practice for:*

Accelerated Weathering Test of Bituminous Materials (**D 529 – 39 T**).

*Standard Specifications for:*

Asphalt Roll Roofing Surfaced With Powdered Talc or Mica (**D 224 – 55**),
Asphalt Shingles Surfaced With Mineral Granules (**D 225 – 54**), and
Asphalt Siding Surfaced With Mineral Granules (**D 699 – 54**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 79 members; 61 members have returned their ballots, of whom 60 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. R. SNOKE,
*Chairman.*

G. W. ROBBINS,
*Secretary.*

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE D-9
## ON
# ELECTRICAL INSULATING MATERIALS*

Committee D-9 held three meetings during the year: in Atlantic City, N. J. during the Annual Meeting of the Society; in St. Louis, Mo., on October 17, 18 and 19, 1956; and in Roanoke, Va., on February 25, 26 and 27, 1957.

The committee consists of 206 members, of whom 98 are classified as producers, 63 as consumers, and 45 as general interest members. During the year deaths of two members occurred: H. L. Curtis, chairman of the committee from 1930 to 1934 and an honorary member, and C. H. Lyman, representative of Standard Oil Co. (California).

Subcommittee II on Radiation Effects was organized as a joint subcommittee with Subcommittee D-20 on Plastics. The title and scope of two subcommittees were expanded. For additional discussion see the section of the report on activities of subcommittees.

Committee D-9 has taken an active part in the work on Electrical Insulating Materials of the International Electrotechnical Commission. Subcommittee IV is participating in the work of TC10 on Insulating Oils. Subcommittee XII has been active in considering proposals from TC15 regarding international standards for electrical insulating materials. Eight members of the committee are working on the various Working Groups of TC15, dealing with standards for Electrical Strength, Electrical Resistivity, Tracking, Voltage Endurance, Dielectric Constant and Loss, Temperature Properties of Insulating Materials, and Conditioning. One member is on the Experts Committee of TC15. Five committee members attended the meeting of TC15 in Munich in June 1956: J. F. Dexter, I. G. Easton, K. N. Mathes, J. R. Perkins, and A. H. Scott.

The custom of holding symposia on topics of interest was continued at the meeting in Roanoke with the presentation of papers on aging tests for transformer oil by members of Subcommittee IV.

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-9 presented the following recommendations to the Society through the Administrative Committee on Standards:

*Tentative Specifications for:*

Silicone Varnished Glass Cloth and Tape (D 1459-57 T),

*Tentative Methods of Testing:*

Fully Cured Silicone Rubber Coated Glass Fabric and Tapes Used for Electrical Insulation (D 1458 - 57 T), and

*Revision of Tentative Methods of Test for:*

Electrical Resistance of Insulating Materials (D 257 - 54 T).

These recommendations were accepted by the Standards Committee on January 31, 1957 and the new and revised tentatives appear in the 1957 Compilation of ASTM Standards on Electrical Insulating Materials.

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* **Presented at the Sixtieth Annual Meeting of the Society, June 16-21, 1957.**

## RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication two Suggested Methods, two new tentatives, revisions of three tentatives, tentative revisions of one standard, the revision of one standard with reversion to tentative, and adoption of one tentative as standard with revision. Details of the above recommendations are covered in the Appendix to this report.[1]

The committee has reviewed all other tentatives under its jurisdiction which have been published for two or more years, and recommends that they be continued in their present status until prospective revisions are made.

The recommendations of this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Insulating Varnishes, Paints, and Lacquers* (W. E. Harvey, chairman) recommended the publication as tentative of the Method for Set Time of Thermosetting Phenol Formaldehyde Varnishes, and adoption as standard with revision of Tentative Methods of Testing Silicone Varnishes (D 1346 - 56 T) as set forth in the Appendix to this report.

Current projects include development of a method of test for gel time, methods of testing high-temperature properties of varnishes by (a) helical coil test at various temperatures, (b) rectangular bar shear test and (c) preparation of free films, and methods for thermal stability of varnishes.

*Subcommittee II on Effects of Radiation* (D. S. Ballantine, chairman) is a joint subcommittee with Committee D-20 on Plastics, and the organization meeting was held on February 28, 1957. Initial organization comprises Section A on Nomenclature and Definitions, Section B on Dosimetry, Section C on Correlation of Radiation Sources, Section D on Effects of Total Dosages and Dose Rate, and Section E on Post Irradiation Effects. Due to considerable overlapping or interrelation of interests among the sections, some reorganization is indicated.

It is expected that initial consideration will be confined to organic materials which are more susceptible to radiation damage than inorganic materials. This will greatly simplify the problems of dosimetry.

*Subcommittee III on Plates, Tubes, Rods, and Molded Materials* (E. A. Russell, chairman) prepared a tentative revision of Standards Methods of Testing Sheet and Plate Materials Used for Electrical Insulation (D 229 - 49), as set forth in the Appendix of this report.

Section B on Statistical Property Values has been organized to establish minimum property values on the materials of interest to the subcommittee. The principles of such studies are outlined in the Symposium on Minimum Property Values of Electrical Insulating Materials (ASTM STP 188).

Other work comprises revision of a draft specification for copper-clad XXXP laminates to differentiate between rolled and electro-deposited copper, a study of electrodes for dielectric strength testing in connection with I.E.C. activities, and development of proposed methods for checking stability properties at elevated temperatures.

*Subcommittee IV on Liquid Insulation* (F. M. Clark, chairman) prepared the new Tentative Method for Determination of 2,6 Ditertiary-Butyl Para-Cresol in

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[1] See p. 346.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

New Electrical Insulating Oils, and a minor revision of the Tentative Recommended Practice for Purchase of Uninhibited Mineral Oil for Use in Transformers and Circuit Breakers (D 1040 – 54 T), as contained in the Appendix to this report.

Under consideration are advancement to standard of Tentative Methods of Testing Electrical Insulating Oils (D 117 – 54 T) and Tentative Method of Test for Insulation Resistivity of Electrical Insulating Oil of Petroleum Origin (D 1169 – 54 T).

A suggested method for determination of oxidation stability of transformer oil by oxygen absorption has been drafted. This method has been used with satisfaction by several laboratories.

A symposium on transformer oil, consisting of the papers listed below, was held at the meeting in Roanoke:

"The Use of Solid and Liquid Catalysts for the Accelerated Aging Testing of Transformer Oil," by E. L. Raab, General Electric Co., Pittsfield, Mass.

"Evaluation of Laboratory Tests as Indicators of the Service Life of Uninhibited Electrical Insulating Oils," by T. A. McConnell, Detroit Edison Co., Detroit, Mich.

Liaison continues with other technical groups in the field of liquid insulation, as summarized in the 1956 Annual Report.

*Subcommittee VI on Solid Filling and Treating Compounds* (W. R. Dohan, chairman) is preparing a revision of Tentative Methods of Testing Hydrocarbon Waxes Used for Electrical Insulation (D 1168 – 54 T), and new methods of test for polymerizable embedding materials.

*Subcommittee VII on Insulating Fabrics* (F. P. West, chairman) recommended the revision of Tentative Methods of Testing Pressure-Sensitive Tapes Used for Electrical Insulation (D 1000 – 55 T) and revision and reversion to tentative of Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 48) as set forth in the Appendix to this report. The new specification and method accepted during the year by the Administration Committee on Standards had been prepared by the subcommittee.

*Subcommittee VIII on Insulating Papers* (H. A. Anderson, chairman) concurs in action by ASTM Headquarters to assign responsibility for preparation of specifications for aluminum foil for capacitors to Committee B-7, rather than to organize a new committee covering all capacitor materials.

Statistical methods are being applied to evaluation of interlaboratory test data on insulating paper of chlorides, pH, and aqueous extract conductivity. Results indicate an acceptable degree of uniformity in the analytical results reported by eleven cooperating laboratories.

The Proposed Specifications for Electrical Insulating Paper Sulfate (Kraft) Tissue for Capacitor Dielectric[3] permit subjecting the capacitor tissue to an accelerated life test, "as agreed upon between the manufacturer and purchaser." Data from round-robin accelerated life tests made by six laboratories indicate a controllable degree of variation among laboratories, and show the practicability of the test procedure for incorporation in the proposed specifications.

*Subcommittee IX on Mica Products* (A. H. Scott, chairman) recommended the revision of Tentative Specification for Natural Muscovite Mica Based on Visual Quality (D 351 – 53 T), as appended to this report. This is the culmination of several years of intensive work by experts in the field. As the exact qualities for any class are a matter of opinion, it has been extremely difficult to get agreement on all points in the twelve classes. A set of visual standards to supplement the verbal descriptions of the various classes has been prepared and approved and is available for inspection at ASTM Headquarters. Subcommittee IX has agreed to review matched sets prepared by comparison with the visual standards and give them its approval if in the opinion of a group of experts selected for the purpose, they match the standard set.

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[3] 1957 Compilation of ASTM Standards on Electrical Insulating Materials.

A set of standards for waviness has also been prepared and is in the custody of Headquarters. Intensive efforts are being made to devise a method of measuring waviness. The National Bureau of Standards, under the sponsorship of the General Services Administration, has devised two methods, one of which was considered to have special merit and will be developed further.

*Subcommittee X on Magnet Wire Insulation* (K. N. Mathes, chairman) was organized in 1956. Initial efforts were devoted to classification of work within its scope and to an effective organization to carry on work. Meetings have been held separately from meetings of the main committee, and the next subcommittee meeting will be held in the fall of 1957.

At a meeting on January 30, 1957 at ASTM Headquarters, consideration was given to development of test methods for resistance to Freon, oil and askarel contamination, moisture resistance, and varnish and compound compatibility. NEMA activities on magnet wire insulation were reviewed. Topics included methods for measuring insulation thickness on magnet wire, cut through and thermal plasticity, abrasion, solderability, and bond test. Pending clarification of the problems, action has been deferred on test methods for edge coverage of rectangular wire, of smoothness of coating, and concentricity of coating.

*Subcommittee XI on Nomenclature, Significance, and Statistics* (C. L. Craig, chairman) expanded its scope during the year to include responsibility for review and approval, from the standpoint of adequacy and format, of statements on the usefulness of tests, including scope, definition, and significance, and for recommending statistical practices and standard nomenclature. The subcommittee has reviewed and approved scope, significance and definition statements in new and revised standards sponsored by five other subcommittees.

New sections on nomenclature and on statistical practices are compiling a glossary of terms, and developing a recommended practice for sampling.

The Symposium on Minimum Property Values held in the spring of 1956 has been published as STP No. 188. A new symposium on "Application of Statistics" will be presented at the fall 1957 meeting, under the chairmanship of C. J. Photiadis.

*Subcommittee XII on Electrical Tests* (N. W. Edgerton, chairman) is sponsoring publication of two suggested methods, including a statement to precede all such methods, which are appended to this report.

The subcommittee also sponsored the recommendation accepted by the Administrative Committee on Standards for revision of Tentative Method of Test for Electrical Resistance of Insulating Materials (D 257 – 54 T).

The subcommittee believes there is a need for tests of the type covered by the two Suggested Methods; and hopes that publication will encourage trial and comment, leading eventually to recommendations for publication as tentative. The Suggested Method offers a means of obtaining experience and data; and of making a promising procedure available for trial at an earlier date than if publication is deferred until tentative status is justifiable. The subcommittee is preparing two more Suggested Methods: "The Liquid Immersion Method for Measuring Dielectric Constant and Loss of Polyethylene" and a "Method for Determining the Resistance to Corona of Solid Electrical Insulating Materials."

Other activities include evaluation of electrostatic phenomena encountered as static surface charges on insulating materials; coordination of tests on insulating materials of such things as thermal capability, corona resistance, electrode pressure in dielectric breakdown tests, and dielectric loss of low-loss materials and of thin films, and of procedures for measuring very high insulation resistance.

*Subcommittee XIV on Conditioning* (G. M. Armstrong, chairman) is considering suggestions to revise the Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 54) to include a system of descriptive nomenclature similar to that used in MIL-P specifications for specifying conditioning procedures and to clarify the significance of tolerances for humidity and temperature. The subcommittee is cooperating in development of international conditioning standards in committees of the International Organization for Standardization and the International Electrotechnical Commission. The work of this subcommittee is a joint activity with Committee D-20 on Plastics.

*Subcommittee XV on Technical Papers and Research* (L. B. Schofield, chairman), formerly the Special Subcommittee on Technical Papers, has expanded its scope to include research. Research activity will be of an advisory nature on the suitability of proposed research type studies for reference to the other subcommittees.

One symposium was held during the year on transformer oil under the direction of Subcommittee IV. Scheduled for the Fall of 1957 is a symposium on "Application of Statistics."

Two symposia were published during the year as ASTM Special Technical Publications: No. 188, Minimum Property Values of Electrical Insulating Materials and No. 198, Symposium on Corona.

This report has been submitted to letter ballot of the committee, which consists of 206 members; 110 members returned their ballots, of whom 107 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

H. K. GRAVES,
*Chairman.*

J. E. GIBBONS,
*Secretary.*

## APPENDIX

## RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given the recommendations affecting certain standards covering electrical insulating materials referred to earlier in this report. The standards and tentatives appear in their present form in the 1955 Book of ASTM Standards, Part 6.

### Suggested Methods Published as Information

The committee recommends for publication as information only the following two suggested methods appended hereto.[1]

*Suggested Methods for:*

Corona Measurement, and
Dust and Fog Tracking Resistance Test for Electrical Insulating Materials.

The committee recommends the following statement to precede all suggested methods which it sponsors:

> This method has been suggested for trial use. Its publication here implies only that it is deemed worthy of such trial. It has not received the evaluation required for a tentative method. Comments and criticisms are solicited, and should be addressed to: American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa.

The committee believes there is a need for tests of this type and that publication as suggested methods makes it possible to get early test data, comments, and criticism, from which a decision to publish as a tentative method can be made sooner than would be otherwise possible.

### New Tentatives

The committee recommends for publication as tentative the following methods and specifications as appended hereto.[2]

Method of Test for Set Time of Thermosetting Phenol Formaldehyde Varnishes. This method is recommended to fill a need for this type of test not heretofore available in ASTM procedures.

Method of Test for 2,6 Ditertiary Butyl-Para-Cresol in New Electrical Insulating Oils. The method provides a procedure, not heretofore available in ASTM methods, for determining the amount of this widely used oxidation inhibitor, also called DBPC, in new electrical oils. The intention is that significance and definition statements will appear in the method by title, which will be referenced to the general Tentative Methods of Testing Electrical Insulating Oils (D 117 – 54 T).

### Revisions of Tentatives

The committee recommends revisions as indicated of the following three tentatives and their continuation as tentative:

Tentative Specification for Natural Muscovite Mica Based on Visual Quality (**D 351 – 53 T**):[3] Revise as appended hereto.[2]

The revision is the result of several years of work by experts in the field, with the object of reconciling conflicting opinions.

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[1] The suggested methods appear in the compilation of "ASTM Standards on Electrical Insulating Materials," September, 1957.

[2] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 6.

[3] **1955 Book of ASTM Standards, Part 6.**

Tentative Methods of Testing Pressure-Sensitive Tapes Used for Electrical Insulation (**D 1000 – 55 T**):[3] Revise as appended hereto.[2]

New sections are being added on breaking strength, elongation, and length which will increase the usefulness of the method.

Tentative Recommended Practice for Purchase of Uninhibited Mineral Oil for Use in Transformers and Circuit Breakers (**D 1040 – 54 T**):[3]

*Table I.*—Add the following:

| Property | Specified Value | ASTM Method |
|---|---|---|
| Inorganic chlorides and sulfates................ | None | D 878 |

The addition is a conventional inclusion in commercial specifications for insulating oils.

## TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as follows of Standard Methods of Testing Sheet and Plate Materials Used for Electrical Insulation (**D 229 – 49**).[3] The revision is intended to apply only to means of measuring insulation resistance of laminated thermosetting insulating materials, when tested parallel to the laminations under conditions of high humidity. This type of measurement is not provided for in existing ASTM Methods.

*New Sections.*—Add the following new Sections 32 to 37 and renumber the subsequent sections accordingly:

### INSULATION RESISTANCE

32. *Scope.*—This method is intended to apply only to laminated thermosetting sheet materials when tested parallel to the laminations under conditions of high humidity.

33. *Definition.*—The insulation resistance of a laminated thermosetting sheet material is the ratio of the direct voltage applied to two electrodes inserted in the specimen to the total current between them. It is the value of resistance, in megohms, obtained when tests are performed parallel to laminations in accordance with the conditions of this method.

34. *Significance.*—(*a*) This test method is of value in determining the suitability of a material for use as electrical insulation under severe service conditions where high humidities may be encountered. The higher the insulation resistance the better is the quality of the material as an insulator for direct voltages when used under conditions simulating those of the test.

(*b*) As to suitability for certain electrical applications, thermosetting laminated sheet materials may be classified broadly by this insulation resistance test into groups differing from each other approximately by powers of 10. Although the test is usually not sufficiently reproducible to permit grading materials closely within a particular classification, it is suitable for use in research and development, in manufacturing control, and in establishing typical values.

35. *Test Specimens and Electrodes.*—(*a*) Four specimens of each material shall be tested, unless otherwise specified.

(*b*) For materials about $\frac{1}{32}$ in. or more in thickness, the specimens shall be rectangular in shape, with dimensions 2 in. by 3 in. by the thickness of the material, and they shall be provided with tapered pin electrodes as shown in Fig. 3 of the Methods of Test for Electrical Resistance of Insulating Materials (ASTM Designation: D 257).

(*c*) For materials less than about $\frac{1}{32}$ in. thick, the specimens and electrode arrangements shown in Fig. 1 and Fig. 2 of Method D 257 may be employed. When this is done, it should be so indicated in the report.

NOTE.—Insulation resistance values obtained with binding post electrodes (Fig. 1 of Method D 257) or strip electrodes (Fig. 2 of Method D 257) are not directly comparable with those obtained with tapered pins (Fig. 3 of Method D 257).

(*d*) Before insertion or application of electrodes each specimen shall be cleaned (unless otherwise specified or agreed upon) in accordance with The Tentative Recommended Practice for Cleaning Plastic Specimens for Insulation Resistance Testing (ASTM Designation: D 1371). Specimens should be handled by touching only the edges. Nylon, rayon, or surgical rubber gloves are recommended as a precaution against possible contamination of the specimens.

36. *Procedure.*—(*a*) The insulation resistance determination shall be made in accordance with Tentative Methods of Test for Electrical Re-

sistance of Insulating Materials (ASTM Designation: D 257).

(*b*) Properly mount the specimens (See Section 15 of Method D 257) and condition for 96 ± 2 hr at 35 ± 2 C, 90 ± 2 per cent RH, in accordance with Procedure C of The Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (ASTM Designation: D 618).

NOTE 1.—Great care must be exercised in measuring and controlling the relative humidity and temperature in the test chamber within the limits specified, since the insulation resistance is extremely sensitive to humidity and temperature. For information, see The Recommended Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions (ASTM Designation: E 104).

NOTE 2.—In mounting the specimens for measurement, it is important that there shall be no conductive paths between the measuring terminals other than those through and over the surfaces of the specimens between the electrodes.

(*c*) At the end of the conditioning period and with the specimens still in the conditioning chamber, measure the insulation resistance of each specimen, employing an electrification time of 1 min and an applied d-c potential of 500 v as specified in Section 4 (*a*) of Method D 257.

37. *Report.*—The report shall include the following:

(*1*) The name, grade, etc., of each material tested.

(*2*) The thickness and other dimensions of the specimen.

(*3*) The type of electrode arrangement employed, that is, whether as in Fig. 1, Fig. 2, or Fig. 3 of Method D 257.

(*4*) The relative humidity and temperature in the conditioning chamber.

(*5*) The insulation resistance of each specimen and the average insulation resistance of each material tested, in megohms.

## REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (**D 350 - 48**)[3] be revised by the adoption of the tentative revisions[3] and with this revision the standard be reverted to tentative status.

## ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Methods of Testing Silicone Varnishes (**D 1346 - 56 T**),[4] be approved for reference to letter ballot of the Society for adoption as standard with the following editorial change:

*Section 21.*—In the second sentence, change "$\frac{1}{2}$ in." to read "$1\frac{1}{2}$ in." Add the following Note to the end of the Section.

NOTE.—Excessive weave shift or yarn displacement in ECC-B $1\frac{1}{2}$ in. glass tape can affect dielectric breakdown results on varnished tape specimens. For this reason, care must be taken to use uncoated glass tape which has not been distorted.

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[4] 1956 Supplement to Book of ASTM Standards, Part 6.

# REPORT OF COMMITTEE D-11
## ON
# RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-like Materials held two meetings during the year: in Atlantic City, N. J. on June 20 to 22, 1956, and in Philadelphia, Pa. on February 6 to 8 , 1957. Numerous subcommittees met in conjunction with the main committee. The SAE-ASTM Technical Committee on Automotive Rubber and its several sections has met on a quarterly basis.

The ISO Technical Committee 45 on Rubber has accepted the invitation of the United States to meet in this country in November 1959. The next meeting of ISO/TC 45 will be held in Zurich, Switzerland, in September, 1957, and several members of the American Group are expected to attend.

The Society, through Committee D-11, is cooperating with the Rubber Division of the American Chemical Society and the Rubber and Plastics Group of The American Society of Mechanical Engineers in sponsoring an International Rubber Conference in Washington, D. C., during the week of November 8, 1959. Messrs. S. Collier, R. D. Stiehler, H. G. Bimmerman, and J. J. Allen are representing Committee D-11 in making plans for the meeting.

R. F. Anderson of the B. F. Goodrich Co. has succeeded Fred Thorn as chairman of Subcommittee XVI on Packings since Mr. Thorn has retired, and R. G. Seaman has succeeded H. L. Fisher as chairman of Subcommittee VIII on Nomenclature and Definitions due to Dr. Fisher's retirement.

Several new liaison appointments with other technical committees have been made. Howard Tangenberg replaces B. S. Garvey, Jr. on Working Group 13 on Flex Cracking of ISO/TC 45; C. W. Pickells was appointed to ASA Sectional Committee C59 on Electrical Insulating Materials; R. W. Schatzel was appointed to Sectional Committee C8 on Insulated Wire and Cable (Other than Telephone and Telegraph); J. Breckley has accepted appointment as the D-11 representative on Subcommittee 11 on Subsieve Testing of Committee E-1; John Ingmanson was appointed Research Coordinator for Committee D-11 to work with the main ASTM Research Committee.

A. W. Carpenter, who retired in June 1956 as Secretary of Committee D-11, was elected as a permanent member of the Advisory Committee.

H. L. Fisher was elected as an Honorary Member of the committee in recognition of his years of work on the committee and for the outstanding job he did in preparing and editing the Glossary of Terms Relating to Rubber.

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

*New Tentative Method for:*

Strain Testing of Vulcanized Rubber (D 1456 - 57 T),

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* Presented at the Sixtieth Annual Meeting of the Society, June 16-21, 1957.

*Revision of Tentative Methods of:*

Dynamic Testing for Ply Separation and Cracking of Rubber Products (D 430 - 51 T),
Testing Compressed Asbestos Sheet Packing (D 733 - 53 T),
Testing Rubber-Coated Fabrics (D 751 - 52 T),
Test for Resistance of Vulcanized Rubber or Synthetic Elastomers to Crack Growth (D 813 - 52 T),

*Revision of Tentative Specifications and Methods of Test for:*

Concentrated, Ammonia Preserved, Creamed and Centrifuged Natural Rubber Latex (D 1076 - 54 T), and

*Revision and Reversion to Tentative of Standard Method of Test for:*

Weather Resistance Exposure of Automotive Rubber Compounds (D 1171 - 55).

The revision and reversion to tentative of Standard Method D 1171 was accepted by the Standards Committee on February 18, 1957; the other recommendations were accepted on January 31, 1957. They will appear in the April, 1957 Compilation of ASTM Standards on Rubber and Rubber-Like Materials.

On March 6, 1957 the following recommendations were accepted by the Administrative Committee on Standards:

*New Tentative Method of Test for:*

Change in Length of an Elastomeric Vulcanizate Resulting from Immersion in a Liquid (D 1460 - 57 T),

*Revision of Tentative Method of Test for:*

Change in Properties of Elastomeric Vulcanizates Resulting from Immersion in a Liquid (D 471 - 55 T), and

*Revision of Tentative Specification for:*

Elastomer Compounds for Automotive Applications (D 735 - 55 T).

They will appear in the April, 1957 Compilation of ASTM Standards on Rubber and Rubber-Like Materials.

The new Tentative Method for Strain Testing of Vulcanized Rubber (D 1456 - 57 T) provides a procedure for determining the strain of soft vulcanized rubber compounds under a specified stress after the load has been suspended from the specimen for a specified period of time. The revision of the Tentative Method for Dynamic Testing for Ply Separation and Cracking of Rubber Products (D 430) brings this method in line with the ISO/TC 45 recommendations and more in line with current practice. The Tentative Method for Testing Compressed Asbestos Sheet Packing (D 733) was revised to eliminate some internal inconsistencies. The revisions of the Methods of Testing Rubber-Coated Fabrics (D 751) were made to clarify the testing procedures. The revisions of the Method for Crack Growth of Vulcanized Rubber (D 813) were made to bring this specification in line with ISO/TC 45 recommendations and with current practice. The revisions of the Specification and Methods of Test for Concentrated Natural Rubber Latex (D 1076) add two additional types of latex preserved with low ammonia and other preservatives and also revise and clarify the Methods for Volatile Fatty Acids and KOH number. The revision of the Method for Determining the Weather Resistance of Automotive Rubber (D 1171) adds a procedure for the quality rating of exposed specimens.

The new Tentative for Measuring Change in Length of Elastomeric Vulcanizates after Immersion in Liquids provides a method for evaluating the effect of liquids on rubber by observing the change in length of the specimen through the transparent wall of the container while specimen is still immersed. The revision of the Method for Change in Properties of Elastomeric Vulcanizates Resulting from Immersion in a Liquid (D 471) eliminates Method B. This is replaced by the new method mentioned above. The revision of the Specifications for Automotive Rubber Compounds (D 735) changes the elongation and volume swell values for Grade TA 706 in Table V.

## NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Method for Sampling and Sample Preparation for SBR (Styrene-Butadiene) Rubber.

This method provides procedures for sampling and sample preparation for tests of SBR (Styrene-Butadiene Rubber). It includes sample preparation for chemical tests, Mooney viscosity, physical properties, shrinkage determination and volatile content.

Method of Test for Penetration of Hard Rubber by Type D Durometer.

This method has been developed to provide a means of measuring the hardness of hard rubber with the Shore Type D Durometer.

## REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following nine tentatives and their continuation as tentative:

### Method of Sample Preparation for Physical Testing of Rubber Products (D 15 - 55 T):[2] Revise as appended hereto.[1]

The formulas have been rearranged and brought up to date, and mixing procedures have been modified based on actual experience.

### Specifications for Friction Tape for General Use for Electrical Purposes (D 69 - 52 T):[2]

*Section 12.*—Change the second sentence to read, "Each roll shall contain not less than 82½ ft. and the average length of the rolls in any sampling shall not exceed 85 ft."

*Section 13.*—Add the word "average" before "thickness" in the first sentence.

*Section 19.*—Transfer the center heading "Methods of Testing" to precede Section 20; renumber Section 19 as Section 18 and change to read as follows:

18. *Sampling.*—(*a*) Sample rolls shall be selected at random from each shipment in accordance with the following table:

| Number of Rolls in Shipment | Number of Sample Rolls |
|---|---|
| 50 to 200 | 2 |
| 201 to 500 | 3 |
| 501 to 1000 | 4 |
| 1001 to 5000 | 5 |

(*b*) A shipment shall be the amount of material, not to exceed 5000 rolls, shipped or intended for shipment to a customer and covered by one bill of lading.

(*c*) Each sample roll shall be tested for conformance to all requirements of the specifications.

(*d*) At least 2 ft of the outer layer of each sample roll shall be removed and discarded before taking test specimens.

(*e*) The test specimen shall be unwound from the roll at a slow uniform rate without jerking.

*Section 18.*—Renumber as Section 19 and change to read as follows:

19. *Rejection and Retests.*—(*a*) The purchaser reserves the right to reject any part of the shipment that does not comply with Sections 14 and 15.

(*b*) The purchaser reserves the right to reject a shipment from which two or more sample rolls fail to meet the requirements of any individual test or measurement prescribed in these specifications, without additional sampling. If only one sample roll fails an individual test or measurement or the average length of the sample rolls exceeds the prescribed value, a second set of samples shall be taken in accordance with Section 18 (*a*) and all rolls in the second sampling shall be subjected to that test or measurement wherein the failure occurred. The purchaser reserves the right to reject the shipment if the second sampling fails to conform with the applicable requirement.

(*c*) The manufacturer shall replace rejected tape and shall pay freight charges for the return of such material.

*Section 20.*—Change the heading from "Cold Adhesion Test" to read "Adhesion Test."

*Section 26.*—In the last sentence, delete the words "and the maximum, minimum and average of these five measurements shall be reported." Substitute the words "and the average of these five measurements shall be the average thickness referred to in Section 13."

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[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 6.

[2] 1955 Book of ASTM Standards, Part 6.

### Specifications for Rubber Insulating Tape (D 119 - 48 T):[2]

*Section 8.*—In the first sentence add the word "average" before the words "thickness" and "width." In line 4, place a comma after "in." and change the remainder of Section to read, "and the average length of the rolls in any sampling shall be not more than 5 per cent in excess of the nominal length."

*Section 14.*—Transfer the center heading "Methods of Testing" to precede Section 15; renumber Section 14 as Section 13 and change to read as recommended above for the new Section 18 of Specification D 69.

*Section 13.*—Renumber as Section 14 and change to read as recommended above for the new Section 19 of Specification D 69, except in Paragraph (*a*) to refer to Sections 9 and 10, and in Paragraph (*b*) to refer to Section 13 (*a*).

*Section 18* (*a*).—In the first sentence, change "7 in." to read "12 in." and add as the second sentence, "The test specimen shall be marked for these purposes: 2 in. for the initial winding, 5 in. for the actual test portion and the remaining 5 in. for attaching the test weight."

*Section 19.*—In the first sentence, change "7 in." to read "12 in."

*Section 20.*—Delete from the last sentence the words "and the maximum, minimum and average of these five measurements shall be reported." Substitute the words "and the average of these five measurements shall be the average thickness and width referred to in Section 8."

### Methods of Testing Rubber Hose (D 380 - 51 T):[2] Revise as appended hereto.[1]

This method has been completely rewritten to make it consistent with current practice.

### Method of Testing Hard Rubber Products (D 530 - 55 T):[2]

*Section 3* (*g*).—Change to read as follows:

> (*g*) *Heat-Distortion Temperature.*—The heat-distortion temperature is an experimentally determined value obtained by observing the temperature at which an arbitrary distortion of a test specimen is produced by an arbitrary set of test conditions.

*Section 5* (*c*).—Replace the last sentence with the following:

> The specimens normally shall be conditioned for at least 1 hr at (23 ± 2 C) 73.4 ± 3.6 F before testing, and shall be tested at the conditioning temperature. All tests made at other than normal temperature shall be recorded with a notation clearly stating the test temperature.

*Section 31.*—Change to read as follows:

> 31. *Heat-Distortion Temperature.*—The heat-distortion temperature of hard rubber shall be determined in accordance with the Method of Test for Heat-Distortion Temperature of Plastics (ASTM Designation: D 648).

*Sections 32, 33, 34 and 35.*—Delete these sections and renumber the subsequent sections accordingly.

### Method of Testing Automotive Air Brake and Vacuum Brake Hose (D 622 - 55 T):[2]

*Table I.*—Add the following new dimensions for air brake hose:

| Inside Diameter of Hose, in. | Duty Type | Length of Specimen, in. | Dimensions of Form, in. (See Fig. 1) | | | |
|---|---|---|---|---|---|---|
| | | | *A* | *B* | *R* | *C* (min) |
| $\frac{3}{16}$ ..... | .. | 9 | $4\frac{1}{2}$ | 3 | $1\frac{1}{2}$ | $\frac{1}{2}$ |
| $1\frac{3}{32}$ ..... | .. | 10 | $4\frac{3}{4}$ | $3\frac{1}{2}$ | $1\frac{3}{4}$ | $\frac{3}{4}$ |

### Methods of Testing Asphalt Composition Battery Containers (D 639 - 52 T):[2]

*Section 24.*—Change to read as follows:

24. *Nature of Test.*—This test measures the tendency of a battery container to develop external or internal cracks or porosity, or both, as a result of abrupt and severe changes in temperature.

*Section 25.*—Add the following new Paragraph (*c*):

(*c*) *Electrical Measuring Device.*—Two cast lead electrodes ⅜ in. diameter by 6 in. long mounted in either hard rubber or laminate handles. One electrode shall be soldered to the base of a light socket as shown in Fig. 3 (the accompanying Fig. 1), using a polarized receptacle and plug, and one 110-v 15-w lamp.

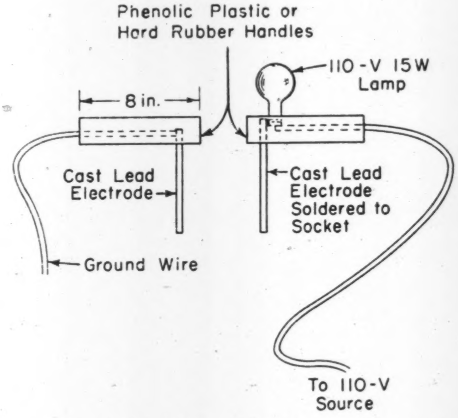
Phenolic Plastic or Hard Rubber Handles
110-V 15W Lamp
8 in.
Cast Lead Electrode
Cast Lead Electrode Soldered to Socket
Ground Wire
To 110-V Source

Fig. 1.—Sketch of Electrical Measuring Device.

*New Figure.*—Add a new Fig. 3 as shown in the accompanying Fig. 1, and renumber the present Fig. 3 as Fig. 4.

*Section 26.*—Change Paragraph (*d*) to read as follows:

(*d*) Visual examination of the container shall first be made to note the development of any cracks or leaks. If no noticeable leakage is found on visual examination, the container is then checked, utilizing the electrical measuring device.

Add the following new Paragraphs (*e*) and (*f*):

(*e*) *Partition Check.*—Test the partitions for leaks by dipping the electrodes into acid on both sides of each partition. If a light shows, wash the top of the partition and the adjacent corners with a wet cloth and wipe dry. Then wash again with alcohol or acetone and wipe dry. If the light persists, a leak is likely. Wash and dry the container thoroughly, and examine the partitions for cracks. If none are visible, cut the container lengthwise and reexamine the partitions. Sometimes only a pinhole or a fine crack in a knit line is responsible. Examination for cracks followed by breaking the partition vertically away from the sides may reveal a crack. A smooth surface in the crack indicates acid penetration. The depth of penetration indicates the weak spot which may go through the partition at some point.

(*f*) *Outside Wall Check.*—Outside wall leaks are clearly visible when the container is removed from the cooling chamber, as a crack will allow a small stream of acid to run down the container. These tend to close up on warming. A wet area may indicate a very small leak or porous spot. Test with the lamp by putting one electrode in acid in the adjacent cell and touching the end of the other electrode to the wet spot or to the top of the solid stream of acid. A very small leak may not light the lamp. Often only a slight puff of smoke or steam will result at the end of the electrode on the outside of the container. Repeated testing at this point may not show a failure, as the leak may be too small to continue passing current. If the container has a rough or irregular outside surface it may not be possible to get sufficient current to light the lamp.

Note.—In testing either partitions or the outer wall, if a leak is indicated by the test lamp, the container should be emptied, washed, dried, and the suspected area cut or broken out for observation. If the suspected area does not show evidence of acid penetration, the test apparatus and procedure should be reviewed to make certain failures are not due to an acid film on the surface that conducts a current and tends to give false results.

### Specifications for Heavy-Duty Neoprene Sheath for Wire and Cable (D 752 – 54 T):[2]

*Title.*—Change to read, "Specifications for Heavy-Duty Black Neoprene Sheath for Wire and Cable."

*Section 1* (*a*).—In the first sentence after "Vulcanized" add the word, "black."

### Specifications for Light-Duty Neoprene Sheath for Wire and Cable (D 753 – 54 T):[2]

*Title.*—Change to read, "Specifications for General Purpose Neoprene Sheath for Wire and Cable."

*Section 1 (a).*—In the first line, add "general purpose" before the word vulcanized.

*Table I.*—Change the original tensile strength from "1200 psi" to read 1500 psi, change the tensile strength after air oven test from "1000 psi" to read 1250 psi, and change the tensile strength after oxygen pressure test from "1000 psi" to read "1250 psi."

### Method of Test for Viscosity of Rubber and Rubber-Like Materials by the Shearing Disk Viscometer (D 927 – 55 T):[2]

*Table I.*—In the Section on method of preparation add to the end of the first paragraph, "Measure the viscosity of standard rubber compounds prepared in accordance with ASTM Method D 15 – 57 T between 2 and 4 hr after mixing." In the second paragraph, change "between 110 and 130 F" to read "122 ± 9 F."

Change the last two sentences to read "Prepare two specimens $1\frac{3}{4}$ in. in diameter from the thickest portion of the sheeted sample. Allow the massed rubber to rest at room temperature for at least 30 min before measuring its viscosity."

### Specifications for Ozone Resisting Rubber Insulating Tape (D 1373 – 55 T):[2]

*Section 9.*—Change as recommended above for Section 8 of Specifications D 119.

*Section 15.*—Transfer the center heading "Methods of Testing" to precede Section 16, renumber Section 15 as Section 14 and change to read as recommended above for the new Section 18 of Specifications D 69.

*Section 15.*—Renumber as Section 14 and change to read as recommended above for the new Section 19 of Specifications D 69, except in Paragraph (*a*) to refer to Sections 10 and 11, and in Paragraph (*b*) to refer to Section 14 (*a*).

*Section 19.*—Change to read as follows:

19. (*a*) *Fusion and Tackiness Tests.*—A specimen 12 in. in length shall be taken from the roll, care being observed not to touch the surface to be tested with the hands or otherwise. The test specimen shall be marked for these purposes: Two inches for the initial windup, five inches for the actual test portion, and the remaining five inches for attaching the test weight. Two inches of the specimen shall be wound on the mandrel, and then a weight of 3 pounds per inch of width of tape shall be attached to the end of the specimen. Should the weight be insufficient to elongate the tape 200 per cent or more, additional downward force shall be applied by hand until 200 per cent elongation has been produced. The tape shall then be wound on the mandrel at a uniform rate of 12 inches per minute. The weight shall then be removed and the specimen allowed to stand for 10 minutes. If the clamp, used to attach the weight to the specimen, weighs less than 4 oz. per inch of width, it may remain attached to the specimen during this waiting period. During this period the tape shall show no tendency to unwind from the mandrel. The weight shall then be attached again and the tape allowed to unwind slowly. The mandrel shall revolve not more than two and one-half turns in the first 2 minutes, during which time the tape shall sustain the weight or shall break at the point of unwinding.

*Section 21.*—Delete this section and renumber Section 22 as Section 21.

*Section 22.*—Renumber as Section 21, and in the last sentence delete "and the maximum, minimum and average of these five measurements shall be reported." Substitute the words "and the average of these five measurements shall be the average thickness and width referred to in Section 9."

### Method for Testing Synthetic Rubber Latices (Butadiene-Styrene Copolymers) (D 1417 – 56 T):[3]

*Section 5 (b).* Change the note to read as follows:

NOTE.—An Alternative Method for Determination of Total Solids in Synthetic Rubber Latex is described in ASTM D 1076 – 54 T for Natural Rubber Latex.

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[3] 1956 Supplement to Book of ASTM Standards, Part 6.

### EDITORIAL CHANGES

The committee recommends the following editorial changes as indicated in the following Standards:

**Method of Test for Tension Testing of Vulcanized Rubber (D 412 – 51 T).**[2]

*Section 3 (e).*—Add the following note after the third paragraph:

NOTE.—Extensive investigation in January 1957 revealed that most users of ring test specimens were using a single spool on each clamp with the lower spool being power rotated.

**Standard Method of Test for Viscosity and Total Solids Content of Rubber Cements (D 553 – 42):**[2]

*Section 4.*—Add the following note to the end of this section:

NOTE.—An alternative method for the determination of viscosity is described in ASTM D 1084 – 55 T.

### REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[1] of the Standard Method of Test for Resistance to Light Checking and Cracking of Rubber Compounds (**D 518 – 44**)[2] and reversion of the method to tentative status.

An additional Method C has been prepared covering the use of tapered specimens. The balance of the method also includes some revisions of wording.

### ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (**D 865 – 54 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

### TENTATIVES CONTINUED WITHOUT REVISION

All the remaining tentatives have been reviewed by the subcommittees and since many are under consideration for revision, they are recommended for continuation as tentative.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Mechanical Rubber Hose* (R. A. Bonnell, chairman) prepared the revision of D 380 – 51 T referred to earlier in the report.

*Subcommittee V on Insulated Wire and Cable* (J. T. Blake, chairman) is considering the preparation of specifications for silicone rubber insulation; synthetic rubber, heat resisting and moisture resisting insulation; and synthetic rubber moisture resisting performance insulation.

*Subcommittee VI on Packings* (R. F. Anderson, chairman) is carrying out round-robin tests on stress relaxation, and a program for measuring the sealability characteristics of packings. Consideration is being given to a corrosion test procedure for addition to Methods D 733.

*Subcommittee XI on Chemical Analysis of Rubber Products* (W. P. Tyler, chairman) is attacking the problem of standardizing test methods for the purity of rubber chemicals. One paper has been written covering extensive work done by a task group and will be published soon.[5] A Method for Determining Melting Range will result from this work.

*Subcommittee XIII on Synthetic Elastomers* (B. S. Garvey, Jr., chairman) is working on a number of additional test methods for evaluating properties of synthetic rubbers. With the publication of Recommended Practices for the Nomenclature and Description of Types of Styrene-Butadiene Rubbers (SBR)

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[5] Published as ASTM *STP No. 229.*

(D 1418 and D 1419) the question of the procedures for assigning numbers for new types is now under consideration.

*Subcommittee XVI on Classification and Specifications for Rubber Compounds* (J. F. Kerscher, chairman) is working on a basic revision of a method of classification of rubber compounds to make its application broader and more flexible.

*Subcommittee XX on Adhesion Tests* (H. H. Irvin, chairman) is considering revisions of the various methods for adhesion tests to make them applicable to testing at elevated temperatures.

*Subcommittee XXIV on Tests for Coated Fabrics* (K. L. Keene, chairman) is working on several new test methods for coated fabrics including a diaphragm burst testing method and abrasion tests.

*Subcommittee XXV on Low-Temperature Tests* (R. S. Havenhill, chairman) is continuing work on improvements in the low-temperature brittleness tests, hardness test and stiffness test.

*Subcommittee XXIX on Compounding Ingredients* (A. E. Juve, chairman) is working on a proposed method for evaluating carbon blacks.

This report has been submitted to letter ballot of the committee, which consists of 251 members; 179 members returned their ballots, of whom 151 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. COLLIER,
*Chairman.*

JOHN J. ALLEN,
*Secretary.*

# REPORT OF COMMITTEE D-12
## ON
# SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year: on March 11 and 12, 1957, in New York City.

The following subcommittee chairmanship appointments were made: H. R. Suter, Subcommittee G-5 on Program Planning, and J. R. Wiebush, Subcommittee T-3 on Analysis of Dry Cleaning Materials and Subcommittee S-3 on Specifications for Drycleaning. The latter appointment is a replacement for G. P. Fulton, long-time chairman of these subcommittees whose resignation, because of change in emphasis of duties, was regretfully accepted; his seat on the Advisory Committee was taken by W. H. Joy.

Liaison representatives appointed were R. E. Hauber to Committee C-22 on Porcelain Enamel; W. H. Koch to Committee E-2 on Emission Spectroscopy; and L. T. Howells to Committee E-8 on Nomenclature.

The committee consists of 117 members, of whom 56 are classified as producers, 33 as consumers, and 28 as general interest members, with 4 consulting members.

### NEW TENTATIVE

The committee recommends for publication as tentative a Method of Test for Copper in Soaps and Soap Products, as appended hereto.[1]

### REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specification for Trisodium Phosphate (**D 538 - 55 T**):[2]

*Table I.*—Revise as follows:

In line 3, change "$PO_2O_5$" to read "$P_2O_5$," correcting an apparent typographical error.

In line 5, total alkalinity for Type I max, change "39" to read "40;" for Type II max, change "35" to read "37."

In line 6, phosphoric anhydride for Type I min, change "42" to read "39.5;" for Type II min, change "38" to read "36."

### REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as follows of the Standard Definitions of Terms Relating to Soaps and Other Detergents (**D 459 - 56**)[3] and reversion of the definitions to tentative:

Add the following new definitions:

*Precision.*—The degree of agreement of repeated measurements of the same property, expressed in terms of dispersion of test results about the arithmetical mean result obtained by repetitive testing of a homogeneous sample under specified conditions. The precision of a method is expressed quantitatively as the standard deviation computed from the results of a series of controlled determinations.

*Syndet.*—A contraction for the term synthetic detergent used loosely to signify synthetic detergents or compositions containing synthetic detergents.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 7.

[2] 1955 Book of ASTM Standards, Part 7.

[3] 1956 Supplement to Book of ASTM Standards, Part 7.

*Surfactant.*—A contraction for the term surface-active agent.

*Ampholytic Surfactant or Amphoteric Surfactant.*—A surface-active agent capable of forming, in aqueous solution, surface-active anions or surface-active cations depending on the pH.

## ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentative methods be approved for reference to letter ballot of the Society for adoption as standard without revision:

*Tentative Specifications for:*

Soda Ash (**D 458 - 55 T**),[2]
Sodium Metasilicate (**D 537 - 55 T**),[2]

*Tentative Method of:*

Test for Buffering Action of Metal Cleaners (**D 1279 - 53 T**),[2]
Total Immersion Corrosion Test of Soak Tank Metal Cleaners (**D 1280 - 53 T**),[2]
Test for Rinsing Properties of Metal Cleaners (**D 1281 - 53 T**),[2] and
Aerated Total Immersion Corrosion Test for Metal Cleaners (**D 1374 - 55 T**).[2]

## REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Methods of Sampling and Chemical Analysis of Alkaline Detergents (**D 501 - 55**),[2] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

*Section 29.*—In the second line following "12 $H_2O$," add "$Na_3PO_4 \cdot H_2O$, or $Na_3PO_4$."

*Section 30(b).*—In the first line, following "Weigh 5 g of" add "dodecahydrate, 2.4 g of monohydrate, or 2.2 g of the anhydrous."

*Section 30(c).*—Change to read as follows:

(*c*) *Calculations.*—Calculate the percentage phosphate as follows:

$Na_3PO_4 \cdot 12\ H_2O$, per cent

$$= \frac{\text{wt. of } Mg_2P_2O_7 \times 3415.61}{W}$$

$Na_3PO_4 \cdot H_2O$, per cent

$$= \frac{\text{wt. of } Mg_2P_2O_7 \times 1634.97}{W}$$

$Na_3PO_4$, per cent

$$= \frac{\text{wt. of } Mg_2P_2O_7 \times 1473.26}{W}$$

$P_2O_5$, per cent

$$= \frac{\text{wt. of } Mg_2P_2O_7 \times 637.72}{W}$$

where $W$ in all cases is the weight of the original sample.

## EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

**Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 54):**[2]

*Section 42(d).*—In the last sentence, change "1.318" to read "1.308," and change footnote *6b* to read:

> [6b] The molar ratio can be calculated using the factor 1.317.

**Standard Methods of Chemical Analysis of Soaps Containing Synthetic Detergents (D 820 - 46):**[2]

*Section 15(f).*—Add to the end of this Section "(weight basis)."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

## SUBCOMMITTEE ACTIVITIES

*Subcommittee T-2 on Analysis of Soaps and Synthetic Detergents* (J. C. Harris, chairman):

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Task Group 2 on Rapid Spectrophotometric Determination of Copper in Soap* (E. W. Blank, chairman) prepared the proposed Tentative Method of Test for Copper in Soap, referred to earlier in the report.

*Task Group 3 on Bibliographical Abstracts on Analytical Methods for Surfactants* (J. C. Harris, chairman).—Abstracts issued as *Special Technical Publication No. 150-A* have been supplemented by references in mimeographed form distributed to the committee for interim use.

*Task Group 6 on Volumetric Method of Total Phosphorus Pentoxide* (R. C. Stillman, chairman) is attempting to resolve some differences on a method which has been cooperatively tested.

*Task Group 7 on Glycerol in Soap by Periodate Oxidation* (C. A. Snell, chairman) has distributed samples for cooperative testing of the method.

*Task Group 8 on Determination of Carboxymethyl Cellulose* (J. L. Darragh, chairman) has completed a survey of methods; samples are available, and cooperative tests are to be undertaken.

*Task Group 10 on Sulfate Content in Presence of Phosphates* (H. C. Bennett, chairman) has made further analyses to define more clearly interferences and a cooperative evaluation of an improved test method will be made.

*Task Group 11 on Determination of Active Ingredient by Ultraviolet Absorption* (E. W. Blank, chairman) has completed initial work and a tentative method is being drafted.

*Task Group 14 on Rapid Determination of Moisture by Infrared Lamp* (O. L. Sherburne, chairman). — Cooperative evaluation of a procedure and equipment is under way.

*Task Group 15 on Determination of Active Ingredient in Soaps Containing Synthetic Detergents, by Cationic Titration* (L. E. Weeks, chairman) has completed cooperative studies that demonstrated the necessity for standardization of the cationic reagent used in the analysis of synthetic detergents. The group is investigating selection of a cationic reagent or anionic standard of proven purity.

*Task Group 20 on Total Active Ingredient* (C. A. Cohen, chairman) is developing a method for analysis of alkylbenzene sulfonate.

*Task Group 21 on Characterization of Detergent Alkylate* (J. C. Harris, chairman) has agreed upon the following procedures for the characterization:

*Flash Point:* Method D 92 – 52, Cleveland open cup.

*Water or Moisture:* Method D 1364 – 55 T, Fischer reagent titration method.

*Specific Gravity:* Method D 891 – 51, Specific gravity balance, hydrometer, pycnometer or vacuum pycnometer.

*Saybolt Color:* Method D 156 – 53 T, Saybolt chromometer.

*Doctor Test:* Method D 484 – 52.

*Viscosity:* Method D 445 – 53 T, Kinematic viscosity.

*Aniline Point:* Method D 611 – 55 T.

*Kauri Butanol:* Method D 1133 – 54 T.

*Bromine Number:* Method D 1158 – 55 T, but increase the sample size to 50 g and use 0.1 *N* reducing agent.

*Sediment:* Method to be a visual one, the sampling to be investigated.

*Refractive Index:* Method D 1218 – 52 T, using Bausch & Lomb or other comparable instrument reading to fourth decimal place.

The following methods will be investigated:

*Distillation Range:* Subgroup A (J. L. Darragh, chairman) will contact Committee D-2 on Petroleum Products for the most up to date method.

*Molecular Weight:* Subgroup B (C. A. Cohen, chairman) will investigate refractivity intercept method using measurements of kinematic viscosity, density, and refractive index all at 20 C.

*Acid Wash Color:* Subgroup C (R. C. Stillman, chairman) has the assignment of developing a satisfactory method.

*Standard Sulfonation Procedure and Analysis of Product:* Subgroup D (C. A. Cohen, chairman) will develop a procedure and application of method of analysis.

*Subcommittee T-4 on Analysis of Alkaline Detergents* (W. H. Koch, chairman) has reviewed reports of the task force chairmen M. V. Trexler on the cobalt complex and C. F. Callis on the chromatographic methods of analysis. The subcommittee will evaluate a third method involving ion exchange for the analysis of tripolyphosphate before making any recommendations.

*Subcommittee T-5 on Physical Testing* (M. G. Kramer, chairman).

*Task Group 1 on Methods for Measuring Soil Redeposition* (A. R. Martin, chairman) prepared the Bibliographical Abstracts on Redeposition of Soil on Cotton Fabric published as *Special Technical Publication No. 173.* A cooperative test program was conducted, and the data are to be analyzed.

*Task Group 3 on Measurement of Reflectance of Test Fabrics* (R. B. Diaz, chairman) has reviewed the data from a preliminary test program and is planning further work based on the results.

*Task Group 5 on Brightener Evaluation* (O. C. Bacon, chairman) has conducted cooperative tests, results of which indicate need for a standard instrument method for evaluation of brightening agents. Efforts will be made to find such an instrument, and to determine whether other useful information can be derived from the available data.

*Task Group 6 on Methods for Measuring Detergency on Fabric* (E. B. Ashcraft, chairman) will survey the literature to select pertinent useful methods and will investigate what other organizations are doing work on this subject.

Two new task groups that have been established are *Task Group 10 on Evaluation of Detergents in Machine Dishwashing* (E. V. Plock, chairman), and *Task Group 11 on Study of Primary Deposition of Soil* (chairman to be appointed).

*Subcommittee T-6 on Metal Cleaning* (H. A. Kafarski, chairman) has approved a proposed vitreous enamel detergent corrosion test for submittal to the subcommittee for action.

The subcommittee will review the Methods of Chemical Analysis of Industrial Metal Cleaning Compositions (D 800 – 45) to improve and up-date certain sections.

The automotive industries task group is completing development of laboratory and shop methods for evaluating cleaners for power spray washers.

*Subcommittee S-2 on Specifications for Soaps and Synthetic Detergents* (W. H. Joy, chairman) will investigate specifications applicable to synthetic detergents, scouring powders, liquid floor cleaners, and others with a view toward determining whether these are suitable subjects for standardization.

*Subcommittee G-5 on Program Planning.* Two very pertinent papers were presented at the March meeting: "Application of Ion Exchange Chromatography to the Analysis of Commercial Triphosphate" by J. Kish of Colgate-Palmolive Co, and "A New Instrument for Optical Brightener Evaluation" by H. W. Zussman of Geigy Chemical Corp.

*ASTM D-12 Award Subcommittee* (A. M. Schwartz, chairman) has drafted the following rules governing an award by this committee which are to be acted upon by the committee prior to the next annual meeting:

1. This award shall be presented not oftener than once a year for outstanding achievement in the field of soap and detergent technology along one or more of the following lines: (*a*) analysis and standardization, (*b*) quality control and specifications, (*c*) physical testing methods, (*d*) performance testing methods, and (*e*) theory of physical or chemical behavior of soaps and detergents. The contributions for which the award is presented must have been made available as public knowledge through recognized journals, books, government publi-

cations, institutional publications, or talks before scientific and technical societies. The Award Committee shall take ASTM Committee D-12 members into primary consideration.

2. The award shall consist of a certificate in the form of a suitably inscribed scroll.

3. The award shall be administered and the recipient selected by an award subcommittee consisting of three members of Committee D-12, appointed by the chairman of the Advisory Committee for staggered 3-year terms, the senior member serving as chairman. If a member of the Award Committee is nominated, he shall be replaced by an interim appointee made by the chairman.

4. Nominations may be made in writing to the chairman of the award subcommittee or to the chairman of Committee D-12 at any time during the year up to four months before the annual meeting of the committee, but in no case later than November 15. Nominations can be held over for consideration the following year.

5. The award subcommittee shall select and notify the recipient not later than two months before the annual meeting.

6. The presentation shall be made at the annual meeting as part of a program suitable to the occasion.

7. The rules governing the award may be changed by vote of the membership.

This report has been submitted to letter ballot of the committee, which consists of 117 members, 77 members returned their ballots, of whom 75 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

J. C. HARRIS,
*Chairman.*

E. W. COLT,
*Secretary.*

# REPORT OF COMMITTEE D-13

## ON

# TEXTILE MATERIALS*

Committee D-13 on Textile Materials held two meetings during the year: on October 16 to 19, 1956, and on March 19 to 22, 1957, both in New York City, N. Y. The Officers Committee held a meeting in New York on January 11, 1957.

At the October meeting, there was a Paper Session discussing the activities of the ISO Technical Committee 38 on Textiles and the part that Committee D-13 is taking in this international standardization activity.

At the March meeting, the Harold De Witt Smith Memorial Medal for 1957 was presented to William D. Appel, Chief, Textiles Section, National Bureau of Standards. This was the eighth award of the Smith Medal, which is made for outstanding achievement in research on fibers and their utilization, as a testimonial to the memory of the late Harold De Witt Smith who pioneered in the concept of an engineering approach to the evaluation of the properties of textile fibers and their industrial application.

The following appointments of subcommittee officers were made during the year: D. S. Hamby, chairman of Subcommittee B-8 on Yarn Test Methods, to replace Mr. W. R. Marsden (resigned); R. T. Graham, secretary of Subcommittee B-2 on Nomenclature and Definitions, to replace Mr. W. M. Segall (resigned); J. T. Taylor, chairman of Subcommittee A-10 on Non-Woven Fabrics, vice R. H. Carter (resigned).

J. B. Goldberg was appointed a member of the Joint AATCC-ASTM Committee on Textile Test Methods, to replace H. J. Ball, resigned.

The committee records with deep sorrow the passing of its long-time secretary, William H. Whitcomb, who died on August 8, 1956. Mr. Whitcomb served the committee faithfully as its secretary for 26 years from 1930 to 1956. He will be long missed by his many friends and the committee records with appreciation his many contributions to its work during his many years of service.

### Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1956 Annual Meeting, Committee D-13 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Tentative Tolerances for:*

Yarns Spun from Mixed Fibers (D 1454 – 56 T), and

Spun Rayon and Acetate Yarns (D 1453 – 56 T).

These recommendations were accepted by the Standards Committee on September 5, 1956, and the new tentatives appear in the 1956 Supplement to Book of ASTM Standards, Part 7.

### Recommendations Affecting Standards

The committee is presenting for

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

publication 2 proposed methods as information, 5 new tentatives, revisions of 7 tentatives, revision and reversion to tentative of 2 standards, adoption of 6 tentatives as standard, revisions for immediate adoption of 6 standards, withdrawal of 1 tentative, 2 standards, and 1 tentative revision.

The standards and tentatives affected, together with the revisions recommended, are listed in detail in the Appendix.[1]

All other standards and tentatives not specifically referred to are being studied by the respective subcommittees and it is recommended that they be continued in their present status.

The committee recommends that the columns for "Typp" be deleted and those for "Grex" be replaced by the "Tex" system in the Yarn Number Conversion Table now published as information in Appendix XIII of the Compilation of ASTM Standards on Textile Materials, November, 1956, p. 760.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 366 members; 162 members returned their ballots, of whom 160 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. L. WHITTIER,
*Chairman.*

H. A. EHRMAN,
*Secretary.*

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[1] See p. 364.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquaters.

APPENDIX

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## RECOMMENDATIONS AFFECTING STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials. These standards and tentatives appear in their present form in the 1955 Book of ASTM Standards, Part 7, or in the 1956 Supplement, Part 7.

### PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the following proposed methods as appended hereto.[1]

*Proposed Method of Test for:*

Length and Length Distribution of Cotton Fibers by the Short Array Method, and
Estimating Maturity and Weight per Unit Length of Cotton Fibers by the Causticaire Method.

### NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[2]

*Methods of Test for:*

Thermal Transmission of Textile Fabrics and Batting Between Guarded Hot Plate and Cool Atmosphere,
Dimensional Change of Knit Fabrics (to replace methods D 1231 and D 1232),
Tufted Pile Floor Coverings,
Differential Dyeing Behavior of Cotton (see Appendix X of the "Compilation of ASTM Standards on Textile Materials," November, 1956, p. 707), and
Laundering and Dimensional Restorability of Warp Knit Fabrics.

### REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

**Tentative Methods of Quantitative Analysis of Textiles (D 629 – 56 T):**

Revise as appended hereto.[2]

**Tentative Methods of Identification of Fibers in Textiles (D 276 – 56 T):**

*Section 1.*—Add the word Arnel in alphabetical location.

*Section 3.*—Add the following item:

(*s*) *Benzyl Alcohol,* USP grade.

*Section 8(d).*—Add the word Arnel in alphabetical location.

*Table IV.*—Add under Striated, Few, the requirements in the accompanying Table I.

*Table V.*—Add after Acrilan the requirements in the accompanying Table II.

**Tentative Methods of Testing and Tolerances for Cotton Yarns (D 180 – 54 T):**

*Section 2.*—In Paragraphs (*c*) and (*f*) change references to "grex system" to read "tex system."

*Section 14(a).*—Change to read as follows:

14. (*a*) The number of specimens to be taken for the various tests shall be sufficient to yield the following per cent precision with a probability of 0.95:

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The proposed Methods appear in "ASTM Specifications for Textile Materials," December 1957.

[2] The new and revised Tentatives and Standards appear in the 1957 Supplement to Book of ASTM Standards, Part 7.

| | Yarn No. | Single End Break | Skein Break | Ply Twist | Single Twist |
|---|---|---|---|---|---|
| Precision, per cent. | 3.0 | 4.65 | 3.9 | 5.0 | 10.0 |

## Tentative Recommended Practice for Designation of Yarn Construction (D 1244 – 53 T):

Change all references to "grex system" to read "tex system."

TABLE I.
(For inclusion in Table IV of D 276 – 56 T)

| | Zinc Chlorine Iodide Reagent | Millon's Reagent | Iodide-Sulfuric Acid-Glycerol Reagent | Phloroglucinol and Hydrochloric Acid |
|---|---|---|---|---|
| Arnel.................... | yellow$^{+d}$ | .... | yellow-brown | white |

TABLE II.
(For inclusion in Table V of D 276 – 56 T)

| | Cross-Section | Density | Nitric Acid | Acetone | HCl | Sulfuric Acid | NaOCl | Methylene Chloride | NaOH | Benzyl Alcohol[a] |
|---|---|---|---|---|---|---|---|---|---|---|
| Arnel....... | Irregular | 1.3 | + | Softens | − | + | − | + | − | − |

[a] 50 C for 1 hr. All other fibers under the Benzyl Alcohol heading are insoluble except acetate which is soluble.

At the specified probability of 0.95:

$$\text{precision per cent} = \frac{1.960V}{\sqrt{N}}$$

or

$$N = \left(\frac{1.960V}{\text{precision}}\right)^2$$

where:

$V$ = coefficient of variation, and

$N$ = number of specimens.

In many cases the coefficient of variation will be known from earlier tests on similar material and the formula may be applied immediately. If no such data are available it is suggested that the following schedule be used. This will give the required precision if the coefficient of variation turns out to be no higher than that given in the second line of the accompanying Table III. If, on the other hand, the coefficient of variation turns out to be higher than given in the table additional tests should be made. Application of the formula gives the total number of tests necessary, whence the number of additional tests needed can be obtained by subtraction.

NOTE.—See Fig. 5 of the Proposed Recommended Practice for Calculating Number of Tests to be Specified in Determining Average Quality of a Textile Material, for simplified calculations.

TABLE III.

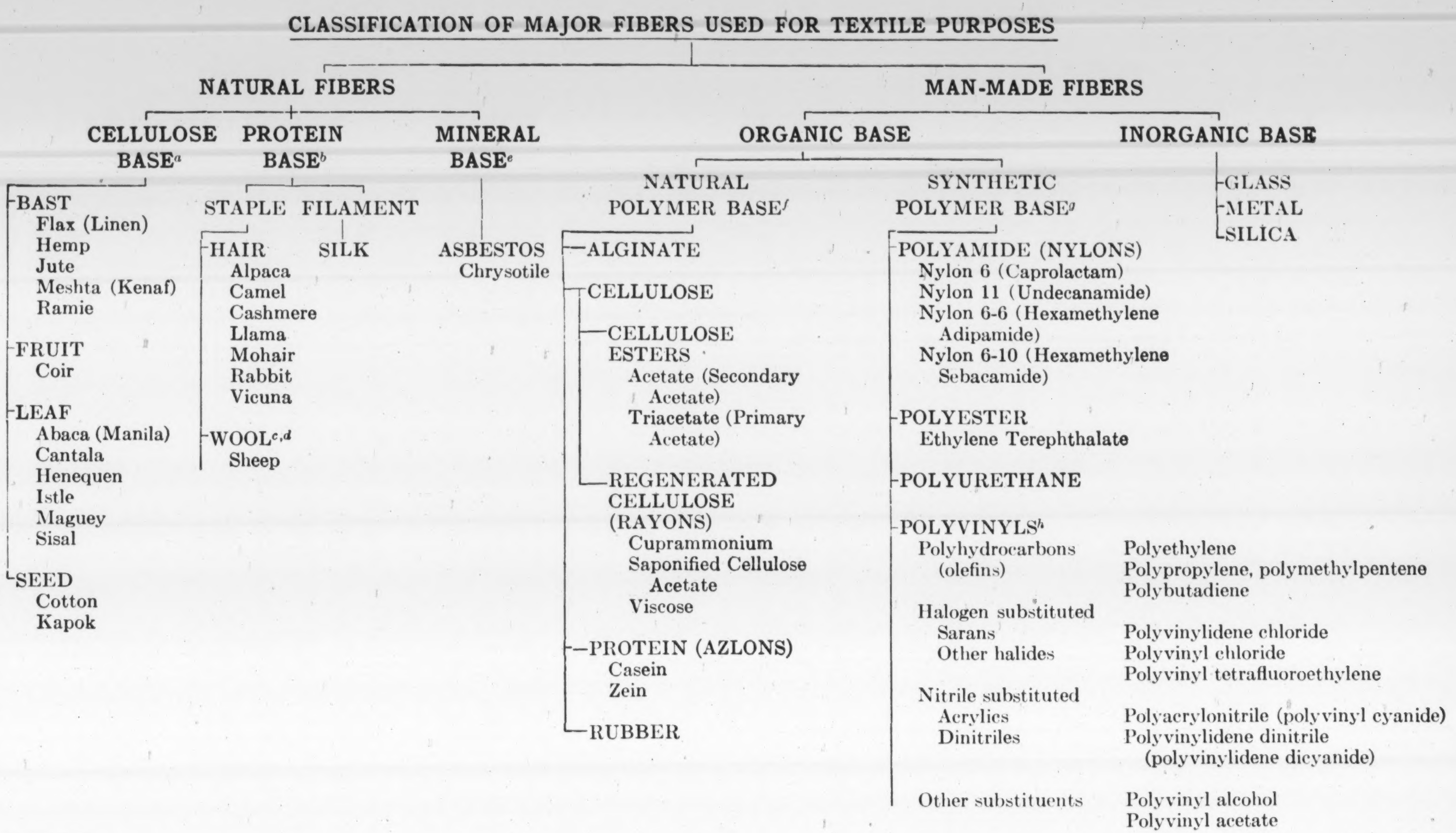
| | Yarn Number | Single End Break | Skein Break | Ply Twist | Single Twist |
|---|---|---|---|---|---|
| Trial number of tests.. | 25 | 40 | 25 | 10 | 15 |
| Illustrative values of coefficient of variation............ | 4.5 | 15.0 | 10.0 | 8.0 | 20.0 |

## Tentative Methods of Testing Spun and Filament Yarns Made Wholly or in Part of Man-Made Organic Base Fibers (D 1380 – 56 T):

Change all references to "grex system" to read "tex system."

*Section 25* (*a*) *and 32* (*a*).—Change to read as follows:

(*a*) The testing machine for strength or extensibility determinations, or both, may be of the constant-rate-of-traverse (pendulum), constant-rate-of-load, or constant-rate-of-specimen-extension types. Such machines shall conform to the Specifications for Textile Testing Machines (ASTM Designation: D 76) with respect to tolerances, load indication, working range and capacity verification, and verification of recorded elongation. Clamps with flat-faced jaws shall be used. They may be operated by cams or pneu-


CLASSIFICATION OF MAJOR FIBERS USED FOR TEXTILE PURPOSES
NATURAL FIBERS
CELLULOSE BASE[a]
BAST
Flax (Linen)
Hemp
Jute
Meshta (Kenaf)
Ramie
FRUIT
Coir
LEAF
Abaca (Manila)
Cantala
Henequen
Istle
Maguey
Sisal
SEED
Cotton
Kapok
PROTEIN BASE[b]
STAPLE
HAIR
Alpaca
Camel
Cashmere
Llama
Mohair
Rabbit
Vicuna
WOOL[c,d]
Sheep
FILAMENT
SILK
MINERAL BASE[e]
ASBESTOS
Chrysotile
MAN-MADE FIBERS
ORGANIC BASE
NATURAL POLYMER BASE[f]
ALGINATE
CELLULOSE
CELLULOSE ESTERS
Acetate (Secondary Acetate)
Triacetate (Primary Acetate)
REGENERATED CELLULOSE (RAYONS)
Cuprammonium
Saponified Cellulose Acetate
Viscose
PROTEIN (AZLONS)
Casein
Zein
RUBBER
SYNTHETIC POLYMER BASE[g]
POLYAMIDE (NYLONS)
Nylon 6 (Caprolactam)
Nylon 11 (Undecanamide)
Nylon 6-6 (Hexamethylene Adipamide)
Nylon 6-10 (Hexamethylene Sebacamide)
POLYESTER
Ethylene Terephthalate
POLYURETHANE
POLYVINYLS[h]
Polyhydrocarbons (olefins)
Polyethylene
Polypropylene, polymethylpentene
Polybutadiene
Halogen substituted
Sarans
Polyvinylidene chloride
Other halides
Polyvinyl chloride
Polyvinyl tetrafluoroethylene
Nitrile substituted
Acrylics
Polyacrylonitrile (polyvinyl cyanide)
Dinitriles
Polyvinylidene dinitrile (polyvinylidene dicyanide)
Other substituents
Polyvinyl alcohol
Polyvinyl acetate
INORGANIC BASE
GLASS
METAL
SILICA

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[a] For more complete information see Definitions D 123, Part B, Table II, C., D., E.

[b] See Part B, Table II, A.

[c] See D 123, Part A, Definitions.

[d] See U. S. Wool Products Labelling Act of 1939.

[e] See Part B, Table II, B.

[f] See D 123, Part B, Table I

[g] Many of these may be found as copolymers.

[h] Most fibers are mixtures or copolymers containing amounts of other compounds in addition to the one named; in general the amounts of other compounds total less than 50 per cent.

Fig. 1.—Textile Fiber Classification Chart.

matically. If, however, (1) the specimens slip through the clamps, or (2) more than 20 per cent of the specimens break between the jaw faces or closer than $\frac{1}{8}$ in. to the nips of the clamps, then snubbing-type clamps shall be used. The snubbing surfaces shall be fixed and integral with the jaw face. They may be circular, having a diameter of not less than 0.5 in., or semi-involute.

**Tentative Methods of Test for Average Fiber Diameter of Wool Tops by the Porous Plug Tester (D 1282 – 53 T):**

Revise as appended hereto.[2]

**Tentative Specifications and Method of Test for Fineness of Mohair Tops (D 1381 – 55 T):**

Revise as appended hereto.[2]

## TENTATIVE REVISION OF STANDARD

The committee recommends the following as a tentative revision of the Standard Definitions of Terms Relating to Textile Materials (**D 123 – 55**):

Add to Part B the Textile Fiber Classification Chart shown in the accompanying Fig. 1.

## REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

**Standard Methods of Testing and Tolerances for Cotton Sewing Threads (D 204 – 56):**

Revise as appended hereto.[2]

**Standard Methods of Testing Rayon and Acetate Staple (D 540 – 44):**

Revise as appended hereto.[2]

## ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

*Tentative Methods of Test for:*

Wool Content of Raw Wool (Laboratory Scale) (**D 584 – 54 T**),
Alkali-Solubility of Wool (**D 1283 – 53 T**),
Strength of Cotton Fibers (Flat Bundle Method) (**D 1445 – 53 T**), and
Maturity of Cotton Fibers (Polarized-Light Method) (**D 1450 – 55 T**).

## ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Magnetic Rating of Asbestos Used for Electrical Purposes (**D 1118 – 50 T**) be approved for reference to letter ballot of the Society for adoption as standard with the following change:

*Section 16.*—Add the following note to this section:

NOTE.—The procedure here described is applicable only under those conditions wherein the magnetic particles being tested exhibit no directional properties or when the particle distribution is such as to give the ultimate in random orientation. The magnetic particles present in chrysotile asbestos, however, are most frequently needle-like or pseudo-fibrous and as such exhibit directional properties with regard to magnetic permeability. Therefore, there must be an understanding as to the extent of the fibrous characteristics and the grain size distribution of the magnetic particles present in the sample under test if a proper evaluation of the resulting magnetic rating is to be truly factual. In view of the fibrous characteristics of the magnetic particles and the resultant directional magnetic properties, the method of sample preparation and the placement of the sample in the sample holder must be carefully pursued in an endeavor to obtain a test specimen that will exhibit a maximum in random orientation.

## REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

## Standard Methods of Testing Felt (D 461 – 53):

*Section 8 (e).*—Change Item (*2*) to read as follows:

(*2*) *Cut Parts.*—The thickness of cut parts which provide a continuous flat felt surface area of 1.129 in. diameter shall be measured as described in Paragraphs (*a*) to (*d*). The thickness of cut parts which do not provide a continuous flat felt surface area of 1.129 in. diameter shall be measured as described in Paragraphs (*a*) to (*d*) by cutting to fit and nesting together a sufficient number of parts to provide continuous felt surface area of 1.129 in. in diameter to which the presser foot can be applied. Cut the parts to be nested so as not to cause excessive compression of the part and, after cutting, condition them in accordance with Section 3. Do not nest the parts so tightly that they bulge at the cut edges, since this will result in obtaining an erroneous reading.

*New Section.*—Add the following procedure for stiffness of felt as a new Section 14, renumbering the present sections accordingly:

14. *Stiffness:* (*a*) *Scope.*—In this procedure the drape stiffness or bending length, and the flex stiffness or flexural rigidity of felt shall be determined by the Drape-Flex or Cantilever type test as described in Method A of the Methods of Test for Stiffness of Fabrics (ASTM Designation: D 1388), with exceptions as prescribed in the following Paragraphs (*c*), (*d*), and (*e*).

(*b*) *Definitions* shall be in accordance with those given in Methods D 1388.

(*c*) *Test Specimens* shall be as prescribed in Section 6 of Methods D 1388 with the exception that test specimens may be cut longer than 6 in. if needed to obtain a bending length that can be measured. If a specimen longer than 6 in. is used due to stiffness of the fabric, add the extra length of specimen (over 6 in.) to the reading measured on the scale.

(*d*) *Procedure.*—Obtain the weight per unit area of the felt in accordance with Section 9 of these methods.

(*e*) *Calculation.*—Unless otherwise specified, average the four readings obtained from all the specimens cut lengthwise of the felt. Do the same for those cut crosswise of the felt. In some cases it may be of interest to differentiate between the sides of the fabric by averaging those readings made with the face side up separately from those with the reverse side up. If this is done, the direction of bending shall be specified. These averages shall be called length of overhang and shall be expressed in inches. Calculate the drape stiffness (bending length), $c$, and the flex stiffness (flexural rigidity), $G$, as follows:

$$c, \text{ in.} = \frac{\text{length of overhang}}{2}$$

$$G, \text{ in.-lb} = c^3 \times W \times 0.482 \times 10^{-4}$$

where:

$W$ = weight in ounces per square yard of felt.

(*f*) *Report.*—Report the drape or flex stiffness of the lengthwise and crosswise directions of the felt separately. If an over-all average figure for the felt is required, calculate and report the geometric average of both directions.

*Section 18.*—Change to read as follows:

18. *Microscopic Examination.*—In the determination of wool content (Section 16) all animal fibers are included as wool. If it is desired to detect the presence of, and identify, fibers other than wool (such as other animal fibers, vegetable and synthetic fibers) the felt shall be subjected to microscopic analysis such as that described in the Identification of Fibers in Textiles (ASTM Designation: D 276). In so far as they are useful for qualitative purposes, the chemical methods given in Quantitative Analysis of Textiles (ASTM Designation: D 629) may be of help in such identification.

## Standard Methods of Test for Resistance of Textile Materials to Microorganisms (D 684 – 54):

Revise as appended hereto.[2]

## Standard Methods of Test for Evaluating Treated Textiles for Permanence of Resistance to Microorganisms (D 862 – 54):

Revise as appended hereto.[2]

## Standard Method of Test for Yarn Number of Yarn from Fabrics (D 1059 – 56):

Change references to "grex system" to read "tex system."

## Standard Specifications and Methods of Test for Asbestos Lap (D 1061 – 54):

*Section 1.*—Change to read as follows:

1. *Scope.*—These specifications and methods of test apply to asbestos lap, a felted asbestos textile product.

*Section 2.*—Delete this section and renumber the subsequent sections accordingly.

*Section 3.*—Redesignate as Section 8 and add a new Section 2 to read as follows:

2. *Definitions:*

(*a*) *Asbestos Lap* is an asbestos textile product which is identified by (*1*) Style, (*2*) Type, (*3*) Weight (grains per yard).

(*b*) *Asbestos Lap* is supplied in two styles: (*1*) *Style I.*—A ribbon-like formation of felted asbestos fibers. (*2*) *Style II.*—A parallel assemblage of Style I lap.

(*c*) *Asbestos Lap* is supplied in two types: (*1*) ferrous, and (*2*) non-ferrous.

*Section 7.*—Renumber as Section 6, and in Paragraph (*a*), line 3, and Paragraph (*b*), lines 3 and 6, change "cones" to read "packages."

*Section 8.*—Renumber as Section 7 and change to read as follows:

7. *Test Conditions.*—All test specimens shall be brought to standard conditions in accordance with Section 8 prior to testing and all tests shall be conducted under standard conditions.

### Standard Methods of Testing Bonded Fabrics (D 1117 – 53):

*Title.*—Change to read, "Methods of Testing Nonwoven Fabrics."

*Section 1.*—Change to read as follows:

1. *Scope:* (*a*) These methods of test are applicable to fabrics which are neither woven, knitted, spun, nor made by conventional wool felting processes, but built up by the interlocking of fibers by chemical bonding agents or of fusible fibers. This is done by mechanical work, chemical action, moisture, and heat. Nonwoven fabrics may be produced by the bonding action of any one or a combination of these factors. The fabric may be made of any fibrous material that can be processed by one of the above methods or any combination of them. Within the above scope, a nonwoven fabric may be defined as a textile structure consisting of a web or mat of fibers held together with a bonding material.

*Section 2.*—In Paragraphs (*a*), (*b*), and (*c*), change "bonded" to read "nonwoven."

*New Sections.*—Add the following new sections:

8. *Tearing Strength* (*Trapezoid Method*).—The tearing strength of nonwoven fabrics shall be determined by the method given in Section 15 (*c*) of the Methods of Testing Woven Fabrics (ASTM Designation: D 39). The words "machine direction" and "cross-machine direction" should be substituted for "warp" and "filling," respectively.

9. *Recovery from Creasing* (*Vertical Strip Method*).—The test methods for woven fabrics as given in the Methods of Test for Recovery of Textile Fabrics from Creasing, Using the Vertical Strip Method (ASTM Designation: D 1295) shall be used. The words "machine direction" and "across-machine direction" should be substituted for "warp" and "filling," respectively.

10. *Air Permeability.*—The air permeability of nonwoven fabrics shall be determined by the Method of Test for Air Permeability of Textile Fabrics (ASTM Designation: D 737), except that in testing light-weight nonwoven fabrics, it may be necessary to support them to prevent sagging. A suitable support is a 14 by 10-count sized gauze, whose unsized weight is less than 200 grains per square yard.

11. *Absorbency Time and Absorptive Capacity.*—The context of Method D 1378 – 55 T, except as modified below, will be included here under the new designation.

*Section 3*(*a*).—Delete "5 cm high, 3 cm in diameter" and substitute "8 cm high, 5 cm in diameter."

*Section 4.*—Change "5 g" to "5 ± 0.1 g."

*Section 13* (*c*).—Delete "on a balance calibrated to 0.1 g" and substitute "to the nearest 0.1 g."

### WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Method of Test for Absorbency Time and Absorptive Capacity of Nonwoven Fabrics (**D 1378 – 55 T**) since it has been recommended for incorporation in Methods D 1117 – 53.

### WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards since they have been recommended for replacement by the proposed new Tentative Method of Test for Dimensional Change of Knit Fabrics:

*Standard Methods of Test for:*

Shrinkage in Laundering of Knit Cotton Fabrics (**D 1231 - 54**), and
Shrinkage in Laundering of Knit Rayon Fabrics (**D 1232 - 54**).

## WITHDRAWAL OF TENTATIVE REVISIO

The committee recommends the withdrawal of the tentative revision submitted in June, 1950, of Section 2 of the Standard Methods of Testing Rayon and Acetate Staple (**D 540 - 44**), as this has been taken into consideration in the complete revision of Methods D 540 being submitted herewith.

# REPORT OF COMMITTEE D-14

## ON

## ADHESIVES*

Committee D-14 on Adhesives held two meetings during the year: at Washington, D. C., on October 11 and 12, 1956, and at Wright Air Development Center, Dayton, O., on April 3 and 4, 1957. On April 5 the committee visited the adhesive testing facilities for aircraft at the Center.

The committee consists of 120 members, of whom 45 are classified as producers, 62 as consumers, and 22 as general interest members.

A new subcommittee on Wood Adhesives is being established.

### NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

*Methods of Test for:*

Amylaceous Matter in Adhesives,
Non-Volatile Content of Aqueous Adhesives, and
Non-Volatile Content of Urea-Formaldehyde Resin Adhesives.

### ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

*Methods of Test for:*

Effect of Mold Contamination on Permanence of Adhesive Preparations and Adhesive Bonds (**D 1286 - 53 T**),[2]
Cross-Lap Specimens for Tensile Properties of Adhesives (**D 1344 - 54 T**),[2]

*Recommended Practices for Determining:*

Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives (**D 904 - 46 T**),[2] and
Strength Development of Adhesive Bonds (**D 1144 - 51 T**).[2]

### TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

*Methods of Test for:*

Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (**D 1002 - 53 T**),
Consistency of Adhesives (**D 1084 - 55 T**),
Resistance of Adhesives for Wood to Cyclic Accelerated Service Conditions (**D 1183 - 55 T**),
Adhesives Relative to Their Use as Electrical Insulation (**D 1304 - 54 T**),
Susceptibility of Dry Adhesive Films to Attack by Roaches (**D 1382 - 55 T**), and
Susceptibility of Dry Adhesive Films to Attack by Laboratory Rats (**D 1383 - 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Strength Properties* (C. E. Britt, chairman) reports progress toward developing a recommended practice for creep testing metal-to-metal adhesives, a recommended practice for determining the suitability of chambers used to conduct extreme-temperature exposure and tests on adhesive bonded lap-shear specimens, and a climbing drum peel test for adhesives. A proposed T-type peel test is also being investigated.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 7.

[2] 1955 Book of ASTM Standards, Part 7.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The rate of tension loading for the shear method D 1002 is being studied in a cooperative program and statistical analysis of resulting data is planned.

A section on stress concentrations is being organized and the extent of interest explored.

In addition to metal bonds, the subcommittee is also concerned with other adherends such as wood and combinations of dissimilar adherends.

*Subcommittee II on Analytical Tests* (G. F. Lipsey, chairman) is developing methods to measure the hydrogen ion concentration of dry adhesive films; to determine the non-volatile content of phenol, resorcinol, and melamine adhesives; and to determine the filler content of adhesives.

The subcommittee is also developing methods for specific gravity and non-volatile content of solvent-type resin adhesives.

*Subcommittee III on Tests for Permanency* (D. A. George, chairman) is continuing a literature survey of information on laboratory-controlled exposure and service tests of adhesives. The test methods on effects of continuous exposure to temperature and humidities are in the process of revision to cover tests at higher temperatures and tests for measuring effects of various aircraft fluids and chemical reagents. A method for determining the permanence of adhesives exposed to natural molds is being written.

Growing interest in the effects of high temperatures and nuclear radiation on the permanence of adhesive bonds may lead to a project in this area.

*Subcommittee IV on Working Properties* (N. J. DeLollis, chairman) is reviewing the various methods of measuring consistency which are in common use in evaluation of materials other than adhesives. The objective is to standardize methods which would measure consistencies not now covered by methods under the committee's jurisdiction.

Two methods for measuring tack and a method for measuring slippage and flow are in various stages of preparation.

Publications on the Interchemical Tackmeter and on a penetration method are being drafted.

*Subcommittee V on Specifications* (S. L. Adams, chairman) has developed test methods for water absorptiveness of labels, for bonding permanency of adhesives used for labeling glass bottles, and a specification for adhesives for automatic machine labeling of glass bottles.

In the packaging field, the subcommittee is developing test methods for the bonding permanency of adhesives for sealing top flaps of shipping cases, for the selection of boxboard samples for the bonding permanency test, and a specification for polyvinyl acetate resin-emulsion adhesives.

A specification for adhesives for acoustical tile has been developed in cooperation with Committee C-20 on Acoustical Materials. Other projects include development of a specification for general purpose (mending) adhesives, and a procedure for sampling adhesives.

*Subcommittee VI on Nomenclature* (G. W. Koehn, chairman) has defined the terms thinner, plasticizer, and penetration and is preparing definitions pertinent to adhesives for paper and to test methods being developed for the terms:

body, consistency, flow, modifier, shortness, solids content, stringiness, retrogradation, tack, and webbing.

*Subcommittee VII on Research Problems* (J. E. Rutzler, Jr., chairman) has sponsored technical sessions on tack, the theory of adhesions, and the behavior of adhesives under test. A task group studying the nature of tack submitted a tentative definition of the term "tack" and a proposal concerning a new method for measuring this property of an adhesive. The task group also surveyed the various known methods for determining tackiness.

Papers presented during meetings of the committee included: "Some Fundamental Principles Derived from Studies of Bonded Joints in Sandwich Construction," by (Mrs.) H. R. Merriman; "A Summary of the Theory and Measurement of Tack," by F. H. Wetzel; "The Effect of Additives on Gap-filling Properties," by A. A. Marra; and "Theoretical Evaluation of the Proportion of the Surface Involved and the Strength of Bonding," by J. E. Rutzler, Jr.

The subcommittee is surveying the literature and will evaluate currently available methods for detecting and measuring stress concentrations in adhesive bonds.

*Subcommittee IX on Editorial Review* (F. W. Reinhart, chairman).—This subcommittee was activated during the year and has 6 members. A method of operation was prepared, approved, and put into effect. Several proposed methods of test and specifications were reviewed and edited. It is planned to edit all documents prior to subcommittee letter ballots.

This report has been submitted to letter ballot by the committee, which consists of 120 voting members; 72 members returned their ballots, of whom 65 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. F. Blomquist,
*Chairman.*

J. J. Lamb,
*Secretary.*

# REPORT OF COMMITTEE D-15

ON

# ENGINE ANTIFREEZES*

Committee D-15 on Engine Antifreezes held two meetings during the year: in Detroit, Mich., on November 15, 1956, and in Washington, D. C. on April 26 and 27, 1957.

The committee regrets the loss of H. R. Wolf, organizer of Committee D-15 and chairman until 1956. Mr. Wolf was the recipient of the ASTM Award of Merit and a special citation from members of Committee D-15. With the passing of Mr. Wolf on January 8, 1957, the committee lost an able counselor and friend.

There have been six resignations from the committee during the past year. Among these was the Secretary, Carl Graham, whose excellent and faithful work on the committee for many years will be missed. Six new members were added to the committee. The committee is composed of 36 members, of whom 10 are classified as producers, 16 as consumers, and 10 as general interest members.

At the April 26 meeting R. E. Vogel was appointed chairman to fill the remaining term of J. M. Clark, and C. O. Durbin was appointed secretary to fill the remaining term of C. F. Graham.

## TENTATIVES CONTINUED WITHOUT REVISION

Committee D-15 recommends that the following tentative specifications and methods be continued as tentative without revision:

*Tentative Specification for:*

Hydrometer-Thermometer Field Tests for Engine Antifreezes (**D 1124 – 53 T**).

*Tentative Method of Test for:*

Ash Content of Concentrated Engine Antifreezes (**D 1119 – 53 T**),

pH of Concentrated Engine Antifreezes (**D 1287–53 T**), and

Glassware Corrosion Tests for Engine Antifreezes (**D 1384 – 55 T**).

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee II on Antifreeze Field Testers* (F. G. Church, chairman) is revising the Tentative Specification for the Hydrometer-Thermometer Field Tester for Engine Antifreezes (D 1124 – 53 T) to include a test method for obtaining basic data with hydrometers for the initial calibration of these testers for primary use by antifreeze producers and tester manufacturers. A collaborative laboratory test program to establish the accuracy and adequacy of the test method has been completed. A Study Group for Special Testers (J. J. Ondrejcin, chairman) is investigating other than hydrometer type testers, such as the direct-freeze type in which a sample of the antifreeze is frozen with dry ice or expanded $CO_2$ gas, and the automatic temperature compensating type.

*Subcommittee IV on Chemical Properties* (J. J. Ondrejcin, chairman).—The Method of Test for Water in Concentrated Engine Antifreezes by the Iodine Reagent Method (D 1123 – 54) is being revised to replace the methanol base reagent with the glycol ether-base reagent as used in the Method of Test for Water in Lacquer Solvents and Diluents (Fischer Reagent Titration Method) (D 1364 - 55 T). Further work will be done on the revisions of this method, particularly in connection with interference by the corrosion inhibitors used in antifreezes. Work has begun to establish a precision section in the Method of Test of pH of Concentrated Engine Antifreezes (D 1287 - 55 T).

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

*Subcommittee VI on Simulated and Actual Service Testing* (C. H. Sweatt, chairman).—The Study Group on Glassware Test Methods (E. L. Gibson, chairman) has completed the first series of cooperative tests on a laboratory screening method for foaming tendency. The need for several modifications was indicated and a second collaborative test program is in progress in which a number of reference coolants are being evaluated by the revised procedure.

The newly formed Study Group on Service Simulation Test Methods (V. O. Hatch, chairman) is working on the apparatus and operating procedure for a bench-type circulating test that would evaluate corrosion and foaming properties under conditions that more closely simulate actual service. A special panel has made recommendations regarding the type of pump and radiator to be used for this test. When the method is established, a number of laboratories will construct the equipment and evaluate its performance with reference antifreezes.

The Chemical Specialties Manufacturers Association has agreed to stock and distribute the metal specimens needed for the Glassware Corrosion Test. Members of Subcommittee VI will submit data obtained with these "standard" specimens for comparison with earlier results from the standpoints of reproducibility and repeatability.

*Subcommittee VII on Specifications* (B. E. Tiffany, chairman) reviewed the fourth draft of a suggested specification for engine antifreezes. A new Federal Government standard in this field was also reviewed and comments sent to the General Services Administration.

This report has been submitted to letter ballot of the committee, which consists of 37 voting members; 26 members returned their ballots, of whom 25 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. E. VOGEL,
*Chairman.*

C. O. DURBIN,
*Secretary.*

# REPORT OF COMMITTEE D-16
## ON
# INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS*

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials and its subcommittees held meetings on June 19 and 20, 1956, in Atlantic City, N. J., and on February 4 and 5, 1957 in Philadelphia, Pa.

Approval has been received from Committee E-1 on Methods of Testing on a series of solidification point thermometers sponsored by Committee D-16. These thermometers, graduated in 0.1 C, are scaled for 76 mm immersion.

### Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1956 Annual Meeting, Committee D-16 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Specification for Industrial 90 Benzene (D 837 – 50) be revised and reverted to tentative status. This recommendation was accepted by the administrative committee on August 9, 1956, and the revised specification appears in the 1956 Supplement to Book of ASTM Standards, Part 5.

### Proposed Method to be Published as Information

The committee recommends for publication as information only the Proposed Method for the Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene, as appended hereto.[1]

### New Tentatives

The committee recommends for publication as tentative the following methods as appended hereto:[2]

*Method of Test for:*

Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration,
Bromine Index of Aromatic Hydrocarbons by Coulometric Titration, and
Solidification Points of Chemicals.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

### Activities of Subcommittees

*Subcommittee A on Monocyclic Aromatics* (S. S. Kurtz, Jr., chairman) prepared the Proposed Method for the Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene referred to earlier in the report. Cooperative tests have been made to determine isomer distribution of nitration grade xylene. Both ultraviolet and infrared techniques were used. Other activities include a study of test methods and specifications for styrene, a method for determining thiophene and similar compounds in toluene and paraxylene, and methods for determining trace quantities of chloride and carbon disulfide in benzene.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] See p. 378.

[2] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 5.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee B on Polycyclic Aromatics* (M. Mitchell, chairman) has reached agreement on methods for determining color, ash, and solidification point of refined naphthalene. Additional work on acid wash test is under way.

*Subcommittee C on Phenolic Compounds* (D. F. Pontz, chairman) intends to publish for information the modified Karl Fischer Method for determination of water in phenol. The committee is investigating color and water solubility of refined phenol.

*Subcommittee D on Nitrogen Heterocyclics* (W. E. Sisco, chairman) has cooperatively tested methods for determining boiling range and water content of refined pyridine and refined quinoline.

*Subcommittee E on Thermometry* (K. H. Ferber, chairman) prepared the specifications for the series of solidification point thermometers which have been approved by Committee E-1.

*Subcommittee G on Statistical Procedure* (W. J. Tancig, chairman) is working on a simplified procedure for statistical analysis of cooperative test data.

This report has been submitted to letter ballot of the committee, which consists of 56 voting members; 38 members returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. F. GOULD,
*Chairman.*

F. J. POWELL,
*Secretary*

## APPENDIX

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# PROPOSED METHOD FOR CALCULATION OF VOLUME AND WEIGHT OF BENZENE, TOLUENE, AND PARAXYLENE[1, 2]

This is a proposed method and is published as information only. The method has been under study in Research Division II on Measurement and Sampling of Committee D-2, and in Subcommittee A of Committee D-16, and is published here to determine its possible usefulness. No action has been taken by Committee D-2 or Committee D-16. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (*a*) These tables are for use in calculating the weight and volume of benzene, toluene, and paraxylene of high purity. A method is given for calculating the volume at 60 F from an observed volume at *t* F. A method is given for calculating the density in pounds per gallon at 60 F.

(*b*) A procedure for the calculation of density in pounds per gallon at 60 F of materials of lower purity is provided.

### Basic Data

2. (*a*) All calculations are derived from densities furnished by the American Petroleum Institute Research Project 44.[3] The densities tabulated have been converted to the basis of pounds per gallon "in air." The tables are based on data for compounds of the highest purity, but can be used for materials in the range indicated on the tables.

(*b*) The basic data and conversion factors used are given in the Supplement along with the method of computation.

### Tables

3. (*a*) Tables I, II, and III each contain three columns as follows:

(*1*) *Column 1.*—Observed temperature in degrees Fahrenheit.

(*2*) *Column 2.*—Multiplying factor for the reduction to 60 F, specifically the ratio of the volume at 60 F to the volume at *t* F.

(*3*) *Column 3.*—Pounds per U. S. gallon at *t* F in air (see Note 3).

(*b*) The values for the multiplying factors for the reduction of volume to 60 F are essentially correct for all purities above 95 per cent for benzene and toluene (Tables I and II) and above 90 per cent for paraxylene (Table III), assuming the presence of the impurities normally present, which are largely aromatic in nature.

(*c*) The values for pounds per gallon given in these tables could be in error by as much as 0.015 lb per gal for each per cent of impurity, if this impurity is mainly nonaromatic in nature.

NOTE 1.—Densities in pounds per gallon at 60 F are given to one more decimal than at other temperatures. The weight in pounds per gallon

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[1] This proposed method is under the joint jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants and the ASTM Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials.

[2] Published as information, June, 1957.

[3] "Selected Values of Properties of Hydrocarbons and Related Compounds," prepared by American Petroleum Institute Research Project 44 at the Carnegie Institute of Technology, Pittsburgh, Pa.

### TABLE I.—PURE BENZENE.

| Temperature, F 1 | Volume Ratio[a] 2 | Weight, lb per U. S. gal[b] in Air 3 | Temperature, F 1 | Volume Ratio[a] 2 | Weight, lb per U. S. gal[b] in Air 3 |
|---|---|---|---|---|---|
| 40 | 1.0130 | 7.461 | 80 | 0.9868 | 7.268 |
| 41 | 1.0124 | 7.457 | 81 | 0.9861 | 7.263 |
| 42 | 1.0117 | 7.452 | 82 | 0.9854 | 7.258 |
| 43 | 1.0111 | 7.447 | 83 | 0.9848 | 7.253 |
| 44 | 1.0104 | 7.442 | 84 | 0.9841 | 7.248 |
| 45 | 1.0098 | 7.438 | 85 | 0.9834 | 7.243 |
| 46 | 1.0091 | 7.433 | 86 | 0.9828 | 7.238 |
| 47 | 1.0085 | 7.428 | 87 | 0.9821 | 7.233 |
| 48 | 1.0078 | 7.423 | 88 | 0.9814 | 7.228 |
| 49 | 1.0072 | 7.418 | 89 | 0.9808 | 7.224 |
| 50 | 1.0065 | 7.414 | 90 | 0.9801 | 7.219 |
| 51 | 1.0059 | 7.409 | 91 | 0.9794 | 7.214 |
| 52 | 1.0052 | 7.404 | 92 | 0.9787 | 7.209 |
| 53 | 1.0046 | 7.399 | 93 | 0.9781 | 7.204 |
| 54 | 1.0039 | 7.394 | 94 | 0.9774 | 7.199 |
| 55 | 1.0033 | 7.390 | 95 | 0.9767 | 7.194 |
| 56 | 1.0026 | 7.385 | 96 | 0.9761 | 7.189 |
| 57 | 1.0020 | 7.380 | 97 | 0.9754 | 7.184 |
| 58 | 1.0013 | 7.375 | 98 | 0.9747 | 7.179 |
| 59 | 1.0007 | 7.370 | 99 | 0.9740 | 7.174 |
| 60 | 1.0000 | 7.365 | 100 | 0.9734 | 7.169 |
| 61 | 0.9993 | 7.361 | 101 | 0.9727 | 7.164 |
| 62 | 0.9987 | 7.356 | 102 | 0.9720 | 7.159 |
| 63 | 0.9980 | 7.351 | 103 | 0.9713 | 7.154 |
| 64 | 0.9974 | 7.346 | 104 | 0.9706 | 7.149 |
| 65 | 0.9967 | 7.341 | 105 | 0.9700 | 7.144 |
| 66 | 0.9961 | 7.336 | 106 | 0.9693 | 7.139 |
| 67 | 0.9954 | 7.331 | 107 | 0.9686 | 7.134 |
| 68 | 0.9947 | 7.327 | 108 | 0.9679 | 7.129 |
| 69 | 0.9941 | 7.322 | 109 | 0.9672 | 7.124 |
| 70 | 0.9934 | 7.317 | 110 | 0.9666 | 7.119 |
| 71 | 0.9928 | 7.312 | 111 | 0.9659 | 7.114 |
| 72 | 0.9921 | 7.307 | 112 | 0.9652 | 7.109 |
| 73 | 0.9914 | 7.302 | 113 | 0.9645 | 7.104 |
| 74 | 0.9908 | 7.297 | 114 | 0.9638 | 7.099 |
| 75 | 0.9901 | 7.292 | 115 | 0.9632 | 7.094 |
| 76 | 0.9894 | 7.288 | 116 | 0.9625 | 7.089 |
| 77 | 0.9888 | 7.283 | 117 | 0.9618 | 7.084 |
| 78 | 0.9881 | 7.278 | 118 | 0.9611 | 7.078 |
| 79 | 0.9874 | 7.273 | 119 | 0.9604 | 7.073 |
|  |  |  | 120 | 0.9597 | 7.068 |

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[a] Based on API Project 44 data.

[b] Calculated according to the Supplement, see p. 385. Values at 60 F agree with values found in API Project 44.

## TABLE II.—PURE TOLUENE.

| Temperature, F | Volume Ratio[a] | Weight, lb per U. S. gal[b] in Air | Temperature, F | Volume Ratio[a] | Weight, lb per U. S. gal[b] in Air |
|---|---|---|---|---|---|
| *1* | *2* | *3* | *1* | *2* | *3* |
| 20 | 1.0235 | 7.431 | 70 | 0.9941 | 7.217 |
| 21 | 1.0230 | 7.427 | 71 | 0.9935 | 7.213 |
| 22 | 1.0224 | 7.423 | 72 | 0.9929 | 7.209 |
| 23 | 1.0218 | 7.418 | 73 | 0.9923 | 7.204 |
| 24 | 1.0212 | 7.414 | 74 | 0.9918 | 7.200 |
| 25 | 1.0206 | 7.410 | 75 | 0.9912 | 7.196 |
| 26 | 1.0200 | 7.406 | 76 | 0.9906 | 7.192 |
| 27 | 1.0194 | 7.401 | 77 | 0.9900 | 7.187 |
| 28 | 1.0188 | 7.397 | 78 | 0.9894 | 7.183 |
| 29 | 1.0182 | 7.393 | 79 | 0.9888 | 7.179 |
| 30 | 1.0177 | 7.388 | 80 | 0.9882 | 7.175 |
| 31 | 1.0171 | 7.384 | 81 | 0.9876 | 7.170 |
| 32 | 1.0165 | 7.380 | 82 | 0.9870 | 7.166 |
| 33 | 1.0159 | 7.376 | 83 | 0.9865 | 7.162 |
| 34 | 1.0153 | 7.371 | 84 | 0.9859 | 7.157 |
| 35 | 1.0147 | 7.367 | 85 | 0.9853 | 7.153 |
| 36 | 1.0141 | 7.363 | 86 | 0.9847 | 7.149 |
| 37 | 1.0135 | 7.359 | 87 | 0.9841 | 7.145 |
| 38 | 1.0130 | 7.354 | 88 | 0.9835 | 7.140 |
| 39 | 1.0124 | 7.350 | 89 | 0.9829 | 7.136 |
| 40 | 1.0118 | 7.346 | 90 | 0.9823 | 7.132 |
| 41 | 1.0112 | 7.341 | 91 | 0.9818 | 7.127 |
| 42 | 1.0106 | 7.337 | 92 | 0.9812 | 7.123 |
| 43 | 1.0100 | 7.333 | 93 | 0.9806 | 7.119 |
| 44 | 1.0094 | 7.329 | 94 | 0.9800 | 7.115 |
| 45 | 1.0088 | 7.324 | 95 | 0.9794 | 7.110 |
| 46 | 1.0082 | 7.320 | 96 | 0.9788 | 7.106 |
| 47 | 1.0077 | 7.316 | 97 | 0.9782 | 7.102 |
| 48 | 1.0071 | 7.311 | 98 | 0.9776 | 7.097 |
| 49 | 1.0065 | 7.307 | 99 | 0.9770 | 7.093 |
| 50 | 1.0059 | 7.303 | 100 | 0.9765 | 7.089 |
| 51 | 1.0053 | 7.299 | 101 | 0.9759 | 7.085 |
| 52 | 1.0047 | 7.294 | 102 | 0.9753 | 7.080 |
| 53 | 1.0041 | 7.290 | 103 | 0.9747 | 7.076 |
| 54 | 1.0035 | 7.286 | 104 | 0.9741 | 7.072 |
| 55 | 1.0029 | 7.282 | 105 | 0.9735 | 7.068 |
| 56 | 1.0024 | 7.277 | 106 | 0.9729 | 7.063 |
| 57 | 1.0018 | 7.273 | 107 | 0.9723 | 7.059 |
| 58 | 1.0012 | 7.269 | 108 | 0.9717 | 7.055 |
| 59 | 1.0006 | 7.264 | 109 | 0.9712 | 7.050 |
| 60 | 1.0000 | 7.260 | 110 | 0.9706 | 7.046 |
| 61 | 0.9994 | 7.256 | 111 | 0.9700 | 7.042 |
| 62 | 0.9988 | 7.252 | 112 | 0.9694 | 7.038 |
| 63 | 0.9982 | 7.247 | 113 | 0.9688 | 7.033 |
| 64 | 0.9976 | 7.243 | 114 | 0.9682 | 7.029 |
| 65 | 0.9971 | 7.239 | 115 | 0.9676 | 7.025 |
| 66 | 0.9965 | 7.234 | 116 | 0.9670 | 7.020 |
| 67 | 0.9959 | 7.230 | 117 | 0.9664 | 7.016 |
| 68 | 0.9953 | 7.226 | 118 | 0.9659 | 7.012 |
| 69 | 0.9947 | 7.222 | 119 | 0.9653 | 7.008 |
|  |  |  | 120 | 0.9647 | 7.003 |

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[a] Based on API Project 44 data.

[b] Calculated according to the Supplement, see p. 385. Values at 60 F agree with values found in API Project 44.

at other temperatures was obtained by machine computation equivalent to multiplying the volume ratio times the density at 60 F, and allowing for the effect of temperature on the air buoyancy.

## Use of Tables

4. (*a*) *Volume Reduction to 60 F.*—Enter the appropriate table in Column

TABLE III.—PARAXYLENE.

Applicable Range 94 to 100 per cent Purity[a]

| Temperature, F | Volume Ratio[b] | Weight, lb per U.S. gal[c] in Air | Temperature, F | Volume Ratio[b] | Weight, lb per U.S. gal[c] in Air |
|---|---|---|---|---|---|
| *1* | *2* | | *1* | *2* | *3* |
| 55 | 1.0027 | 7.228 | 90 | 0.9834 | 7.089 |
| 56 | 1.0022 | 7.224 | 91 | 0.9828 | 7.085 |
| 57 | 1.0016 | 7.220 | 92 | 0.9823 | 7.081 |
| 58 | 1.0011 | 7.216 | 93 | 0.9817 | 7.077 |
| 59 | 1.0005 | 7.213 | 94 | 0.9812 | 7.073 |
| 60 | 1.0000 | 7.209 | 95 | 0.9806 | 7.069 |
| 61 | 0.9994 | 7.205 | 96 | 0.9801 | 7.065 |
| 62 | 0.9989 | 7.201 | 97 | 0.9795 | 7.061 |
| 63 | 0.9983 | 7.197 | 98 | 0.9789 | 7.056 |
| 64 | 0.9978 | 7.193 | 99 | 0.9784 | 7.052 |
| 65 | 0.9972 | 7.189 | 100 | 0.9778 | 7.048 |
| 66 | 0.9967 | 7.185 | 101 | 0.9773 | 7.044 |
| 67 | 0.9961 | 7.181 | 102 | 0.9767 | 7.040 |
| 68 | 0.9956 | 7.177 | 103 | 0.9761 | 7.036 |
| 69 | 0.9950 | 7.173 | 104 | 0.9756 | 7.032 |
| 70 | 0.9945 | 7.169 | 105 | 0.9750 | 7.028 |
| 71 | 0.9939 | 7.165 | 106 | 0.9745 | 7.024 |
| 72 | 0.9934 | 7.161 | 107 | 0.9739 | 7.020 |
| 73 | 0.9928 | 7.157 | 108 | 0.9733 | 7.016 |
| 74 | 0.9923 | 7.153 | 109 | 0.9728 | 7.012 |
| 75 | 0.9917 | 7.149 | 110 | 0.9722 | 7.008 |
| 76 | 0.9912 | 7.145 | 111 | 0.9716 | 7.004 |
| 77 | 0.9906 | 7.141 | 112 | 0.9711 | 7.000 |
| 78 | 0.9901 | 7.137 | 113 | 0.9705 | 6.996 |
| 79 | 0.9895 | 7.133 | 114 | 0.9700 | 6.992 |
| 80 | 0.9890 | 7.129 | 115 | 0.9694 | 6.988 |
| 81 | 0.9884 | 7.125 | 116 | 0.9688 | 6.984 |
| 82 | 0.9878 | 7.121 | 117 | 0.9683 | 6.980 |
| 83 | 0.9873 | 7.117 | 118 | 0.9677 | 6.975 |
| 84 | 0.9867 | 7.113 | 119 | 0.9671 | 6.971 |
| 85 | 0.9862 | 7.109 | 120 | 0.9666 | 6.967 |
| 86 | 0.9856 | 7.105 | | | |
| 87 | 0.9851 | 7.101 | | | |
| 88 | 0.9845 | 7.097 | | | |
| 89 | 0.9840 | 7.093 | | | |

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[a] The pounds per gallon figures in this table assume that the 0 to 6 per cent of impurity consists of a normal distribution of $C_8$ aromatic hydrocarbons. In case of question in regard to density, an experimental density should be obtained as prescribed in Section 5.

[b] Based on API Project 44 data.

[c] Calculated according to the Supplement, see p. 385. Values at 60 F agree with values found in API Project 44.

*1* of Tables I, II, or III, selecting that temperature to the nearest degree Fahrenheit at which the bulk volume was measured (temperature *t*), and select the corresponding volume reduction factor (ratio) in Column *2*. Multiply the bulk volume measurement at temperature, *t*, by the factor selected from the table.

**Example 1.**—What is the volume at 60 F of a tank car of paraxylene whose volume was measured to be 9280 U.S. gal at a mean temperature of 88.7 F?

Enter Table I at 89.0 F and note that the "volume ratio" is 0.9840. The volume at 60 F is 9280 × 0.9840 = 9131.5 U.S. gal.

(*b*) *Converting Volume to Weight for Pure and Nearly Pure Materials.*—Multiply the volume in gallons at 60 F (5 digits) by the density in pounds per gallon at 60 F.

**Example 2.**—What is the weight of paraxylene whose volume is 9280 U.S. gal at 88.7 F?

See Example 1. The weight is 9131.5 × 7.2086 = 65,825 lb.

NOTE 2.—A possible alternate method of obtaining weight, which is not recommended, is to multiply the volume at *t* F by the density in pounds per gallon at the same temperature (Column *3* of Tables I, II, and III). This computation may lead to slightly different numbers from the computation recommended in Paragraph (*b*) because of rounding of the tabulated numbers, which affects the last digit. In order to eliminate this possible small computational discrepancy, the desirability of eliminating all pounds per gallon figures except those at 60 F from the tables is being considered.

(*c*) *Converting Volume of Impure Benzene and Toluene to Weight.*—Correct the measured bulk volume to 60 F as described in Paragraph (*a*). Determine the density (all weights in vacuum) at 60 F in grams per milliliter as described in Section 5, keeping all values to five digits. Obtain the value for pounds per U.S. gallon in standard air at 60 F, by means of the following equation and round to five digits:

$$a = (b - A) \times 8.34522 \qquad (1)$$

where:

$a$ = pounds per U.S. gallon in standard air at 60 F,
$b$ = grams per milliliter in vacuum at 60 F, and
$A$ = correction factor as given in Table IV.

Multiply the corrected volume by the calculated pounds per U.S. gallon at 60 F value in order to obtain the weight in pounds in air.

TABLE IV.—CONVERTING TO DENSITIES IN STANDARD AIR.

| Density at 60 F, in Vacuum, g per ml | Correction Factor, A, g per ml |
|---|---|
| 0.82 | 0.001098 |
| 0.83 | 0.001097 |
| 0.84 | 0.001095 |
| 0.85 | 0.001094 |
| 0.86 | 0.001092 |
| 0.87 | 0.001091 |
| 0.88 | 0.001090 |
| 0.89 | 0.001088 |
| 0.90 | 0.001087 |

TABLE V.—FACTORS FOR CONVERTING FROM SPECIFIC GRAVITY IN AIR TO APPARENT DENSITY IN AIR IN GRAMS PER MILLILITER (NOTE 3).

| Specific Gravity in Air | Multiply Specific Gravity in Air by This Number to Get Apparent Density |
|---|---|
| 60/60 F or 15.56/15.56 C | 0.99904 at 60 F |
| 68/68 F or 20/20 C | 0.99823 at 68 F |
| 77/77 F or 25/25 C | 0.99708 at 77 F |

**Example 3.**—What is the weight of the contents of a tank car of benzene having a calculated density (Section 5) of 0.87638 g per ml, whose volume was measured to be 9280 U.S. gal at a mean temperature of 88.7 F?

Enter Table I at 89 F and note that the "Factor for Reducing Volume to 60 F" is 0.9808. The volume at 60 F is 9280 × 0.9808 = 9101.8 U.S. gal. From Eq 1: (0.87638–0.001090) (8.34522) = 7.30449 lb per U.S. gal. The weight of benzene in the tank car is then 9101.8 × 7.30449 = 66484 lb.

TABLE VI.—VACUUM CORRECTIONS, APPARENT DENSITY TO DENSITY IN VACUUM.

| Apparent Density $\frac{W}{V}$ | Correction,[a] plus | Apparent Density, $\frac{W}{V}$ | Correction,[a] plus |
|---|---|---|---|
| 0.70 | 0.00036 | 0.85 | 0.00018 |
| 0.71 | 0.00035 | 0.86 | 0.00017 |
| 0.72 | 0.00033 | 0.87 | 0.00016 |
| 0.73 | 0.00032 | 0.88 | 0.00014 |
| 0.74 | 0.00031 | 0.89 | 0.00013 |
| 0.75 | 0.00030 | 0.90 | 0.00012 |
| 0.76 | 0.00029 | 0.91 | 0.00011 |
| 0.77 | 0.00028 | 0.92 | 0.00010 |
| 0.78 | 0.00026 | 0.93 | 0.00009 |
| 0.79 | 0.00025 | 0.94 | 0.00007 |
| 0.80 | 0.00024 | 0.95 | 0.00006 |
| 0.81 | 0.00023 | 0.96 | 0.00005 |
| 0.82 | 0.00022 | 0.97 | 0.00004 |
| 0.83 | 0.00020 | 0.98 | 0.00003 |
| 0.84 | 0.00019 | 0.99 | 0.00001 |

[a] This table applies for all air density values between 0.0011 and 0.0013 g per ml. For air densities outside this range, the vacuum correction, $C$, should be calculated as follows:

$$C = \frac{d_a}{0.99823} \times \left(0.99823 - \frac{W}{V}\right),$$

where:

$C$ = vacuum correction,

$d_a$ = density of air in the balance case in grams per milliliter,

$W$ = weight,

$V$ = volume, and

$\frac{W}{V}$ = apparent density (Note 3).

## Density Calculation

5. (*a*) Density determinations may be carried out by any procedure known to be reliable to four digits. Method D 941, Test for Density and Specific Gravity of Hydrocarbon Liquids by the Lipkin Bicapillary Pycnometer[4] and Method D 1217, Test for Density and Specific Gravity of Liquids by Bingham Pycnometer[4] are suitable and are written to give density completely in vacuum (corrected) which is required for the computation described herein.

(*b*) If the methods described in Methods D 891, Test for Specific Gravity of Industrial Aromatic Products[4] are used, specific gravity in air (Note 3) is first converted to density in grams per milliliter in air by use of the factors given in Table V.

NOTE 3.—"Specific gravity in air" means that all weighings were in air, and no air buoyancy correction was applied. "Apparent density" for the purposes of this test is defined as the density calculated when the pycnometer volume is calibrated with water, weighed in air, and when the sample is weighed in air, and no air buoyancy correction is used for either weighing, even though the density in vacuum of water is used in calculating the volume. "Density in air" for the purpose of this method is the weight per unit volume in vacuum minus the weight of a volume of air equal to the difference between the volume of the sample and the volume of brass weights equivalent to the weight in vacuum of the sample. If it is desired to make a direct experimental determination of "density in air" using a pycnometer, apply the air buoyancy correction to the water weight, calculate the

TABLE VII.—MULTIPLIERS TO CONVERT FROM DENSITY IN VACUUM AT *t* DEGREES TO DENSITY AT 60 F (15.56 C) IN VACUUM.

| Temperature, *t* | Multiply by | | |
|---|---|---|---|
| | Benzene | Toluene | Paraxylene |
| 68 F or 20 C | 1.00529 | 1.00473 | 1.00443 |
| 77 F or 25 C | 1.01136 | 1.01011 | 1.00947 |
| 86 F or 30 C | 1.01753 | 1.01554 | 1.01459 |

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[4] 1955 Book of ASTM Standards, Part 5.

true volume of the pycnometer from the weight of water in vacuum, and then fill the pycnometer with sample, weigh in air, subtract the weight of the pycnometer in air, and apply no air buoyancy correction. This sample weight in air, divided by the true volume in milliliters is the "density in air" at the experimental temperatures. Densities given in the ASTM-IP Petroleum Measurement Tables (ASTM Designation: D 1250)[4] are on this basis.

(*c*) Convert apparent density in grams per milliliter in air to density in vacuum by use of Table VI.

(*d*) Convert density in vacuum at *t* deg to density at 60 F (15.56 C) using the multipliers given in Table VII on interpolated values.

**Example 4.**—The density in vacuum of a benzene sample was determined at 25 C and found to be 0.87095. The coefficient from the table is 1.01136. The density in vacuum at 60 F is 0.87095 × 1.01136 = 0.88084.

(*e*) Calculate pounds per U.S. gallon in air in accordance with Section 4 (*c*).

## SUPPLEMENT

## METHOD OF COMPUTING TABLES

### *Density Values for Hydrocarbons*

A1. (*a*) The five decimal place values for the density of benzene, toluene, and paraxylene given in Table A1 were supplied by the director of the API Research Project 44.[3] The value at 20 C is considered to be exact to the values given, and are those appearing in API Project 44 (Table 5a).[3] The other values are relative to the 20 C values.

(*b*) By means of a least squares solution, quadratic equations of the following type were obtained:

$$D = d_0 + a_1t + a_2t^2 \qquad (2)$$

where:

$D$ = density, in grams per milliliter in vacuum, at $t$ F,
$d_0$ = constant necessary for the computation (not density at 0 F) from Table A2,
$a_1$, $a_2$ = linear and quadratic coefficients from Table A2, and
$t$ = temperature, in degrees Fahrenheit.

### *Reduction of Volume to 60 F*

A2. (*a*) The following equations were used:

$$P = \frac{C(D - d_a)}{1 - (d_a/d_b)} \qquad (3)$$

$$= 8.346431D - 0.010158 \qquad (4)$$

$$\text{Value to 60 F Factors} = \frac{\text{density at } t\text{F}}{\text{density at 60F}} \qquad (5)$$

where:

$P$ = pounds in air per U.S. gallon at $t$ F,
$C$ = multiplier constant for converting grams per milliliter to pounds per U.S. gallon (0.0022046223/0.00026417) = 8.3452207,

TABLE A1.—DENSITIES OF PURE AROMATIC HYDROCARBONS.

| Temperature, deg Cent | Benzene | Toluene | Paraxylene |
|---|---|---|---|
| −10 | ..... | 0.89465 | ..... |
| 0 | ..... | 0.88542 | ..... |
| 10 | 0.88947 | 0.87619 | 0.86968[a] |
| 20[b] | 0.87901 | 0.86696 | 0.86105 |
| 30 | 0.86845 | 0.85773 | 0.85241 |
| 40 | 0.85774 | 0.84850 | 0.84374 |
| 50 | 0.84688 | 0.83927 | 0.83501 |
| 60 | 0.83588 | 0.83004 | 0.82620 |
| 70 | 0.82473 | 0.82081 | 0.81733 |
| 80 | 0.81343 | 0.81158 | 0.80836 |
| 90 | ..... | 0.80235 | 0.79932 |
| 100 | ..... | 0.79312 | 0.79021 |

[a] For the undercooled liquid.
[b] Correspond to values given in Table 5a of API Project 44.

TABLE A2.—COEFFICIENTS FOR EQ 2 BASED ON VALUES OBTAINED FROM THE DIRECTOR OF API PROJECT 44.

| Material | $d_0$ | $a_1$ | $a_2$ |
|---|---|---|---|
| Benzene | 0.917678 | −0.000553346 | −0.0000002212 |
| Toluene | 0.901829 | −0.000512778 | 0.0000000000 |
| Paraxylene | 0.893055 | −0.000463340 | −0.0000001022 |

$D$ = density of material in grams per milliliter at $t$ F (Eq 2)

$d_a$ = density of air in grams per milliliter at 60 F (0.0012170) (Note 4), and

$d_b$ = density of brass in grams per milliliter at 60 F (8.393185) (Note 4).

NOTE 4.—These values are identical with those used in the calculation of the ASTM-IP Petroleum Measurement Tables (ASTM Designation: D 1250).[4]

(*b*) To judge the extent of applicability of values of pure materials to materials of commercial origin, complete tables were made for what were believed to be most probable compositions. Data for the pure compound were obtained from API Project 44.

(*c*) *Accuracy Based on All Assumptions.*—For all purities of benzene, the differences were less than one unit in the last digit for volume ratios given in Table I, and in only about 5 per cent of the cases was it large enough to appear as one unit in the last digit as a result of the rounding-off process. For toluene, the differences were large so that about 35 per cent of the rounded values differed by one unit in the fourth digit shown in Table II, most of these being below 40 F and above 100 F, and for the lower range of purity. For paraxylene, the effect is equivalent to that for benzene.

### *Conversion of Volume to Weight*

A3. (*a*) *Paraxylene.*—The density of paraxylene is essentially constant for purities from 95 to 100 per cent, provided that the paraffinic impurities are within usual commercial tolerances. Accordingly, tables based on the densities of pure paraxylene are also applicable to commercial grades.

(*b*) *Benzene.*—Although the density of commercial benzene may vary from 0.875 to 0.884 at 60 F, the same volume reduction factors are found applicable throughout. For other than pure material, it is necessary to determine the density, preferably at 20 or 25 C (68 or 77 F) in order to find the weight of a shipment whose volume is measured at any temperature in the range of temperatures given in Table I.

(*c*) *Toluene.*—Although the density of commercial toluene may vary from 0.863 to 0.874 at 60 F, the same volume reduction factors are found applicable throughout. For other than pure material, it is necessary to determine the density, preferably at 20 or 25 C (60 or 77 F) in order to find the weight of a shipment whose volume is measured at any temperature in the range given in Table II.

# REPORT OF COMMITTEE D-17

## ON

# NAVAL STORES*

Committee D-17 held one meeting during the year: on June 21, 1956, at Atlantic City, N. J., in connection with the Annual Meeting of the Society.

The committee regrets the loss due to retirement of E. V. Romaine, who by virtue of continuous service was the committee's oldest member.

Responsibility and jurisdiction for the Standard Specifications for Turpentine (D 13 – 51) and the Standard Methods of Sampling and Testing Turpentine (D 233 – 51) have been transferred to Committee D-17 from Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

The chairman continues to serve as the Society representative on a special committee of the Federal Supply Service and U. S. Department of Agriculture working on the revision of Federal Specifications for naval stores products. These specifications include turpentine (TT-T-801), rosin (LLL-R-626), dipentene (TT-D-376), and pine oil (LLL-O-358).

## REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society.

The committee has found that the table of temperature corrections for barometric pressure as given in the Standard Method of Sampling and Testing Turpentine (D 233 – 51) is based on an obsolete correction factor. The committee also found that similar tables are necessary for the standard methods for testing dipentene (D 801 – 48) and pine oil (D 802 – 49). These temperature correction factors were computed by means of the Sydney-Young equation.

TABLE I.—TEMPERATURE CORRECTIONS (DEG CENT ) FOR BAROMETRIC PRESSURE.[a]

| Observed Barometric Pressure, mm | Aneroid Barometer | Mercurial Barometer Temperature of Barometer | | | |
|---|---|---|---|---|---|
| | | 20 C | 25 C | 30 C | 35 C |
| 780...... | −1.04 | −0.91 | −0.88 | −0.84 | −0.81 |
| 770...... | −0.52 | −0.39 | −0.36 | −0.32 | −0.29 |
| 760...... | 0.00 | +0.13 | +0.16 | +0.19 | +0.22 |
| 750...... | +0.52 | +0.65 | +0.68 | +0.71 | +0.74 |
| 740...... | +1.04 | +1.17 | +1.20 | +1.23 | +1.26 |
| 730...... | +1.56 | +1.68 | +1.71 | +1.75 | +1.78 |
| 720...... | +2.08 | +2.20 | +2.23 | +2.26 | +2.29 |
| 710...... | +2.62 | +2.72 | +2.75 | +2.78 | +2.81 |
| 700...... | +3.16 | +3.24 | +3.27 | +3.30 | +3.33 |

[a] These corrections are calculated as follows: The observed barometric pressure is first corrected to what it would be at 0 C, by means of the table in Circular F, Instrument Division, U. S. Weather Bureau. The corrected barometric pressure is then subtracted from 760 mm (or *vice versa* and the difference multiplied by 0.052 C to give the temperature correction shown in the above Table. The correction factor, $C$, of 0.052 C was calculated using the Sydney-Young equation:

$$C = 0.00012\ (760 - P)(273 + T)$$

where:

$P$ = Observed pressure, corrected to 0 C, in mm, and

$T$ = Median boiling temperature for turpentine, 160 C.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

## Standard Methods of Sampling and Testing Turpentine (D 233 – 51):[1]

*Section 12.*—In Paragraph (*a*), line 3 change "0.057 C" to read "0.052 C." In Paragraph (*b*), lines 13 and 16 change "end point" to read "minimum percentage requirement."

In the second line of the Note, change "750 mm" to read "748 mm." In line 7, change "at the end of the distillation" to read "at the minimum percentage requirement."

*Table I.*—Change to read as shown in the accompanying Table I.

## Standard Methods of Testing Dipentene (D 801 – 48):[1]

*Section 9.*—To the end of Paragraph (*b*) add a sentence to read, "Correct all observed temperatures to compensate for variations in barometric pressure, as described in Section 9 (*c*)."

Add a new Paragraph (*c*) to read as follows:

(*c*) *Barometric Correction.*—The distilling temperature of dipentene is affected by 0.054 C for each millimeter variation of the atmospheric pressure. Therefore, the temperatures observed or specified shall be corrected to permit the distillation to be conducted as though the barometer reading, corrected to 0 C, were exactly 760 mm. When about to begin the distillation, observe and record the barometric pressure and the temperature of the barometer. (No temperature correction on barometer reading is necessary for aneroid type barometers.) From Table I determine the proper temperature correction corresponding to these atmospheric conditions, interpolating to the nearest 0.1 C. If the barometric pressure, corrected to 0 C, is below 760 mm, the temperature correction must be added to the temperatures observed and subtracted from the minimum percentage requirement temperature; if above 760 mm, the correction must be subtracted from the temperatures observed and added to the minimum percentage requirement temperature.

NOTE: *Example.*—Suppose the observed barometric pressure is 745 mm at 28 C, and the 5 per cent boiling point is observed to be 172.0 C. From Table I by interpolating, the temperature correction is seen to be 1.0 C. Therefore the corrected 5 per cent boiling point is 172.0 + 1.0 = 173.0 C. Furthermore the temperature observation point at the minimum percentage requirement (usually 188 C at 760 mm) must be altered to the same extent. Since the dipentene is distilling 1.0 C below what it would at normal pressure, distillation must be discontinued at 188.0 − 1.0 = 187.0 C to determine the percentage distilling below 188 C at 760 mm pressure.

TABLE II.—TEMPERATURE CORRECTIONS (DEG CENT) FOR BAROMETRIC PRESSURE.[a]

| Observed Barometric Pressure, mm | Aneroid Barometer | Mercurial Barometer Temperature of Barometer | | | |
|---|---|---|---|---|---|
| | | 20 C | 25 C | 30 C | 35 C |
| 780 . . . . . . | −1.08 | −0.94 | −0.91 | −0.87 | −0.84 |
| 770 . . . . . . | −0.54 | −0.40 | −0.37 | −0.34 | −0.30 |
| 760 . . . . . . | 0.00 | +0.13 | +0.17 | +0.20 | +0.23 |
| 750 . . . . . . | +0.54 | +0.67 | +0.70 | +0.74 | +0.77 |
| 740 . . . . . . | +1.08 | +1.21 | +1.24 | +1.27 | +1.31 |
| 730 . . . . . . | +1.62 | +1.75 | +1.78 | +1.81 | +1.84 |
| 720 . . . . . . | +2.16 | +2.29 | +2.32 | +2.35 | +2.38 |
| 710 . . . . . . | +2.70 | +2.82 | +2.86 | +2.89 | +2.92 |
| 700 . . . . . . | +3.24 | +3.36 | +3.39 | +3.42 | +3.45 |

[a] These corrections are calculated as follows: The observed barometric pressure is first corrected to what it would be at 0 C, by means of the table in Circular F, Instrument Division, U. S. Weather Bureau. The corrected barometric pressure is then subtracted from 760 mm (or *vice versa* and the difference multiplied by 0.054 C to give the temperature correction shown in the above Table. The correction factor, $C$, of 0.054 C was calculated using the Sydney-Young equation:

$$C = 0.00012\,(760 - P)(273 + T)$$

where:

$P$ = Observed pressure, corrected to 0 C, in mm, and

$T$ = Median boiling temperature for dipentene, 180 C.

*New Table.*—Add a new Table I as shown in the accompanying Table II.

## Standard Methods of Sampling and Testing Pine Oil (D 802 – 49):[1]

*Section 7.*—In Paragraph (*b*), the eighth sentence, place a period after the word "requirement" and delete the remainder of the sentence. Add as the ninth sentence, "Correct all observed temperatures to compensate for variations of barometric pressure, as described in Section 7(*c*)."

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[1] 1955 Book of ASTM Standards, Part 4.

Add a new Paragraph (*c*) to read as follows:

(*c*) *Barometric Correction.*—The distilling temperature of pine oil is affected by 0.059 C for each millimeter variation of the atmospheric pressure. Therefore, the distillation temperatures observed or specified shall be corrected to permit the distillation to be conducted as though the barometer reading, corrected to 0 C, were exactly 760 mm. When about to begin the distillation, observe and record the barometric pressure and the temperature of the barometer. (No temperature correction on barometer reading is necessary for aneroid type barometers.) From Table I determine the proper temperature correction corresponding to these atmospheric conditions, interpolating to the nearest 0.1 C. If the barometric pressure, corrected to 0 C, is below 760 mm, the temperature correction must be added to the temperatures observed and subtracted from the minimum percentage requirement temperature; if above 760 mm, the correction must be subtracted from the temperatures observed and added to the minimum percentage requirement temperature.

TABLE III.—TEMPERATURE CORRECTIONS (DEG CENT) FOR BAROMETRIC PRESSURE.[a]

| Observed Barometric Pressure, mm | Aneroid Barometer | Mercurial Barometer Temperature of Barometer | | | |
|---|---|---|---|---|---|
| | | 20 C | 25 C | 30 C | 35 C |
| 780...... | −1.18 | −1.03 | −0.99 | −0.96 | −0.92 |
| 770...... | −0.59 | −0.44 | −0.41 | −0.37 | −0.33 |
| 760...... | 0.00 | +0.15 | +0.18 | +0.22 | +0.25 |
| 750...... | +0.59 | +0.73 | +0.77 | +0.81 | +0.84 |
| 740...... | +1.18 | +1.32 | +1.36 | +1.39 | +1.43 |
| 730...... | +1.77 | +1.91 | +1.94 | +1.98 | +2.01 |
| 720...... | +2.36 | +2.50 | +2.53 | +2.57 | +2.60 |
| 710...... | +2.95 | +3.09 | +3.12 | +3.15 | +3.19 |
| 700...... | +3.54 | +3.67 | +3.71 | +3.74 | +3.77 |

[a] These corrections are calculated as follows: The observed barometric pressure is first corrected to what it would be at 0 C, by means of the table in Circular F, Instrument Division, U. S. Weather Bureau. The corrected barometric pressure is then subtracted from 760 mm (or *vice versa*) and the difference multiplied by 0.059 C. To give the temperature correction shown in the above Table the correction factor, *C*, of 0.059 C was calculated using the Sydney Young equation:

$$C = 0.00012\ (760 - P)(273 + T)$$

where:

$P$ = Observed pressure, corrected to 0 C, in mm, and

$T$ = Median boiling temperature for pine oil, 215 C.

NOTE: *Example.*—Suppose the observed barometric pressure is 747 mm at 28 C, and the 5 per cent boiling point is observed to be 204.0 C. From Table I, by interpolating, the temperature correction is seen to be 1.0 C. Therefore the corrected 5 per cent boiling points is 204.0 + 1.0 = 205.0 C. Furthermore the temperature observation point at the minimum percentage requirement (usually 225 C at 760 mm) must be altered to the same extent. Since the pine oil is distilling 1.0 C below what it would at normal pressure, distillation must be discontinued at 225.0 − 1.0 = 224.0 C to determine the percentage distilling below 225 C at 760 mm pressure.

*New Table.*—Add a new Table I as shown in the accompanying Table III.

## ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions consisting of definitions for dipentene, monocyclic terpenes, and rosin standards of the Standard Definitions of Terms Relating to Naval Stores and Related Products (**D 804 – 52**)[1] be approved for reference to letter ballot of the Society for adoption as standard.

The tentative revision of "rosin standards" when adopted is to replace the first paragraph of the present standard definition for the term.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Softening Point of Rosin* (J. M. Schantz, chairman) is awaiting the outcome of the study in Committee D-4 and Committee E-1 on combining the two ASTM ring-and-ball softening point methods, D 36 – 26 and E 28 – 51 T, into a single, all-purpose method.

*Subcommittee II on Crystallization of Rosin* (S. R. Snider, chairman), is continuing to study the crystallizing tendency of gum rosin, using 170 representative batch samples collected from all the gum processing plants throughout the producing belt, from Georgia to Mississippi, during the 1956 producing season. All the grades from WW to K were included among these samples. As was previously observed, a wide variation was noted in the tendency of these rosins to crystallize from the solution phase, when tested according to the Proposed Method of Test for Crystallization Tendency of Gum Rosin. The acid number and the softening point of a representative proportion of the samples are being determined, to see whether there is any correlation between these properties and the tendency to crystallize.

*Subcommittee IV on Chemical Analysis of Rosin* (R. Herrlinger, chairman) is continuing its study of methods for determining or computing the fatty acid content of tall oil rosins. Four specially prepared samples consisting of refined wood rosin with added known proportions of fatty acid (oleic acid) were used for this study, with five laboratories collaborating. Two methods have been under study for determining the rosin acids, namely, (1) the modified Wolfe method, as used for the determination of rosin acids in tall oils (ASTM Method D 803), and (2) the Linder-Persson method, as published in the January 1957 issue of the *Journal* of the Oil Chemists Society. The results indicated that both methods could be expected to give dependable, comparable results, but somewhat closer agreement was obtained among the collaborators with the modified Wolfe method. Further collaborative work is planned on the Linder-Persson method, with the idea that it will be possible to have it as an alternate method for determining the rosin acids by a visual titration method. The Wolfe method has been adopted by several manufacturers of tall oil rosin for production control work on the daily plant operations.

*Subcommittee V on Tall Oil* (A. Pollak, chairman) has been in collaboration with the task group from the Subcommittee IX on Varnish, of Committee D-1, which is making a study of methods for the determination of rosin or rosin acid content in compounded protective coatings, such as varnish. Similar contact was made with Subcommittee II, Group 5 on Fatty Acids, of Committee D-1, specifically to provide a method for determining rosin acids in tall oil fatty fatty acids used in the formulation of protective coatings.

*Subcommittee VII on Terpene Hydrocarbons and Pine Oil* (R. E. Price, chairman) found that the correction that has been in use for many years on turpentine was in slight disagreement with the correction computed according to the modern Sydney-Young equation as applied to the distillation of light volatile oils in general. Through oversight, temperature corrections were omitted from the ASTM dipentene and pine oil distillation tests. The proper corrections are now being recommended for all three terpene oil distillation methods. Further revisions in ASTM Method D 233 are now being studied and will be ready for presentation in the near future. Consideration is being given to the advantage of using the distillation procedure as now given in ASTM Method D 1078 for the several terpene oils for which a distillation test is specified.

*Subcommittee IX on Definitions* (J. L. Boyer, chairman) has recommended that the tentative revisions of the standard definitions for "dipentene" and "rosin standards" and the new tentative definitions for "mono-cyclic terpenes" be adopted as standard.

This report has been submitted to letter ballot of the committee, which consists of 36 members; 21 members returned their ballots, of whom 20 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

V. E. GROTLISCH,
*Chairman.*

W. A. KIRKLIN,
Secretary.

# REPORT OF COMMITTEE D-18

ON

# SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held two meetings during the year: in Atlantic City, N. J., on June 22, 1956, in conjunction with the Annual Meeting of the Society, and in Los Angeles, Calif., on September 18, 1956, in conjunction with the Second Pacific Area National Meeting of the Society. Executive Committee meetings were held on June 21, 1956, in Atlantic City, N. J., and on January 6, 1957 in Washington, D. C.

A Symposium on Shear Testing of Foundation Soils by the Vane Method, and a general soils session were sponsored by the committee at the 1956 Annual Meeting, and at the West Coast meeting the committee sponsored three general soils sessions. The Vane method Symposium is being published as *ASTM STP No. 193*, and the papers presented at the West Coast Meeting as *ASTM STP No. 206*.

A further session on soils will be held at the 1957 Annual Meeting of the Society.

A meeting of the committee is planned in Mexico City at the University of Mexico the week of December 8, 1957, and a special committee consisting of W. G. Holtz (chairman), T. W. Van Zelst, and E. A. Abdun-Nur has been appointed to make arrangements for the meeting and to secure papers for soils sessions to be held at that time. The meeting is being held jointly with the Sociedad Mexicana de Mecanica de Suelos.

R. K. Bernhard has been appointed as the representative of Committee D-18 on Committee E-10 on Radioisotopes and Radiation Effects, and Joseph Berman has been appointed to Subcommittee 28 on Microscopy of Committee E-1 and to the Joint Committee on Chemical Analysis by Powder Diffraction Methods.

Harold Allen was elected to honorary membership on the committee in recognition of his outstanding work.

The committee consists of 134 individual members and 19 consulting members, of whom 107 are voting members; 21 are classified as producers, 38 as consumers, and 48 as general interest members.

Changes have been made in the By-laws of the committee to limit the length of appointment of consulting members to 2 years with provisions for renewal of their appointment, to increase the membership of the committee from 100 to 110 members, and to require that recommendations on standards be submitted to the membership at least 10 days before the meeting at which they are to be considered.

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards the proposed

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

Tentative Method of Soil Investigation and Sampling by Auger Boring. The method was accepted by the administrative committee on January 14, 1957, and it appears in the 1956 Supplement to Book of ASTM Standards, Part 3, bearing the designation D 1452 - 56 T.

### ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

*Tentative Method of:*

Test for Bearing Capacity of Soil for Static Load on Spread Footings (**D 1194 - 52 T**),[1]

Repetitive Static Load Tests of Soils, for Use in Evaluation and Design of Airport and Highway Pavements (**D 1195 - 52 T**),[1] and

Nonrepetitive Static Load Tests of Soils, for Use in Evaluation and Design of Airport and Highway Pavements (**D 1196 - 52 T**).[1]

### REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

**Standard Method of Test for Moisture-Density Relations of Soil-Cement Mixtures (D 558-44)**:[1] Revise as appended hereto.[2]

**Standard Method of Wetting-and-Drying Test of Compacted Soil-Cement Mixtures (D 559 - 44)**:[1] Revise as appended hereto.[2]

**Standard Method of Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures (D 560 - 44)**:[1] Revise as appended hereto.[2]

**Standard Method of Test for Cement Content of Soil-Cement Mixtures (D 806 - 47)**:[1]

*Section 5.*—Substitute the following Paragraph (*g*) for the present Paragraphs (*g*) and (*h*), and reletter paragraphs (*i*) and (*j*) accordingly:

> (*g*) Carefully open the filter paper and wash the precipitate into the beaker in which the precipitation was effected. Dilute to 200 ml and add 10 ml of $H_2SO_4$ (1:1). Heat the solution just short of boiling, and titrate it with the standard $KMnO_4$ solution (Note 1) to a permanent pink color. Add the filter paper and macerate it. Continue the titration slowly until the pink color is permanent to 10 sec.
>
> NOTE 1.—The temperature of the standard $KMnO_4$ solution should not vary from its standardization temperature so much as to cause a serious error in the determination of CaO. At ordinary room temperatures the volume of pure water changes to the extent of 0.01 to 0.04 per cent for each degree Centigrade, depending on the temperature.

Reletter paragraph (*j*) as (*i*) and change to read as follows:

> (i) *Alternate Titration Procedure.*—Titrate as described in Paragraph (*g*), except that the $KMnO_4$ solution need not be a standard 0.1 *N* solution but the same solution shall be used in titrating all the components. Omit the blank determination described in Paragraph (*h*).

These four methods are also under the jurisdiction of Committee D-4 on Road and Paving Materials, and the revisions have been approved by that committee.

### TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives which have stood for more than two years without revision:

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[1] 1955 Book of ASTM Standards, Part 3.

[2] The revised standards appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

*Tentative Method of:*

Grain Size Analysis of Soils (**D 422 - 54 T**),
Test for Liquid Limit of Soils (**D 423 - 54 T**),
Test for Plastic Limit and Plasticity Index of Soils (**D 424 - 54 T**),
Test for Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (**D 916 - 47 T**),
Testing Soil-Bituminous Mixtures (**D 915 - 47 T**),
Test for Load-Settlement Relationship for Individual Piles (**D 1143 - 50 T**), and

*Tentative Definitions of:*

Terms and Symbols Relating to Soil Mechanics **D 653 - 42 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

## ACTIVITIES OF SUBCOMMITTEES

*Special Committee on Revision of Procedures for Soil Testing* (W. J. Turnbull, chairman) is working on the revised edition of the Procedures for Testing Soils which should be ready for publication by the Society shortly after the 1957 Annual Meeting.

*Special Committee for Policy on Standards* (W. G. Holtz, chairman) in following through on suggestions expressed at the symposium on the subject held at the 1955 Annual Meeting, agreed on three basic policies to be followed in the development of standard methods of test. These policies involve proper and complete scope; more than one test method may be required for a particular determination, and variations in the test procedure to simulate field conditions may be required in which case procedures information as required for proper interpretation of the data shall be reported.

*Subcommittee G-3 on Nomenclature and Definitions* (C. R. Foster, chairman) in cooperation with the American Society of Civil Engineers, has completed the review of terms which had been compiled relating to soil mechanics and foundation engineering, and on the basis of this review revisions will be recommended in the Definitions of Terms and Symbols relating to Soil Mechanics (D 653 - 42 T).

*Subcommittee R-1 on Surface and Subsurface Reconnaissance* (L. E. Gregg, chairman) has appointed task groups to prepare methods or recommended procedures for reconnaissance techniques of air photography (K. B. Woods, chairman), pedologic methods (A. W. Johnson, chairman) and geophysical reconnaissance (G. W. McAlpin, chairman).

*Subcommittee R-2 on Sampling and Related Field Testing for Soil Investigation* (J. O. Osterberg, chairman) prepared the proposed Tentative Method of Thin-Walled Tube Sampling of Soils and the proposed Tentative Method for Penetration and Split-Spoon Sampling of Soils, referred to earlier in the report. These methods had been included in the 1956 preprinted Annual Report but were withdrawn due to negative votes which have now been resolved.

*Subcommittee R-3 on Physical Characteristics of Soils* (E. E. Bauer, chairman) may recommend minor revisions in Methods D 423 and D 424 as a result of a recent survey. A method similar to Method D 698 will be written to cover a compaction test utilizing a 10-lb hammer. A method for a needle-moisture test is being written.

The cooperative research program to determine the best method of test for the maximum and minimum densities of granular soils is continuing.

*Subcommittee R-4 on Physical Properties of Soils* (N. W. McLeod, chairman) will prepare permeability test procedures for soils.

*Subcommittee R-5 on Structural Properties of Soils* (D. M. Burmister, chairman) is considering the preparation of standards for consolidation, unconfined compression, and triaxial shear tests.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee R-8 on Special and Construction Control Tests* (H. F. Clemmer, chairman) is working on a new method for field density of soils by the sand cone method.

*Subcommittee R-11 on Pile Load Bearing Tests* (W. S. Housel, chairman) is working on revisions of Method D 1143 – 50 T, and consideration is being given to test methods for load capacity for batter piles, lateral load capacities for piles, and load capacities for pile groups.

This report has been submitted to letter ballot of the committee, which consists of 107 voting members; 77 members returned their ballots, of whom 69 voted affirmatively and 8 negatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
*Chairman.*

W. G. HOLTZ,
*Secretary.*

### EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards revisions of the Tentative Method of Test for the Load-Settlement Relationship of Individual Piles Under Vertical Axial Load (D 1143 – 50 T). The revisions were accepted by the Standards Committee on December 2, 1957, and the revised method appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

# REPORT OF COMMITTEE D-19

ON

# INDUSTRIAL WATER*

Committee D-19 on Industrial Water and its subdivisions held meetings on June 20 to 22, 1956, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society, and on January 21 to 23, 1957, in Charleston, S. C.

A Symposium on Steam Quality, presented at the 1956 Annual Meeting of the Society, was sponsored by the Joint Research Committee on Boiler Feed Water Studies, now a research committee of The American Society of Mechanical Engineers, and has been published as ASTM STP 192.

A Symposium on Industrial Water and Industrial Waste Water, sponsored by Committee D-19, was presented on September 20, 1956, during the ASTM Second Pacific Area National Meeting, in Los Angeles, Calif. The Symposium is being published as ASTM STP 207. The committee was represented by its officers: Max Hecht, Chairman (represented by C. K. Rice); R. C. Adams, Vice Chairman; F. R. Owens, (represented by J. K. Rice); O. M. Elliott, Secretary; C. K. Rice, Standards Advisor; and L. D. Betz, Chairman, Subcommittee VII on Industrial Waste Water.

A talk entitled "Three is a Vital Number," was presented during the Industrial Water Luncheon at the West Coast meeting by C. K. Rice, Coordinator, Refinery Technical Lab., Gulf Oil Corp., Philadelphia, Pa., and is to be included with the symposium papers.

The 1957 Marburg Lecture, entitled "Your Most Important Raw Material"[1] and having as its subject industrial water, will be presented on June 19, 1957, during the Annual Meeting of the Society by E. P. Partridge, Director, Hall Laboratory Division of Hagan Chemicals and Controls, Inc.

A Symposium on Methods for the Determination of Dissolved Oxygen[2] will be presented on June 20, 1957, at Atlantic City, N. J. during the Annual Meeting of the Society. It is sponsored by the committee and the following papers comprise the symposium:

"A Study of the Accuracy of Methods of Testing for Dissolved Oxygen in High Purity Water," by K. J. Stoffer, Arabian American Oil Co.

"Polarographic Measurement of Dissolved Oxygen," by W. W. Eckenfelder, Jr., and Brother Conrad Burris, Manhattan College.

"The Beckman Oxygen Analyzer," by T. J. Finnegan and R. C. Tucker, Niagara Mohawk Power Co.

"The Evaluation of the Hartmann and Braun Dissolved Oxygen Recorder," by A. J. Rastaino and A. A. Dominick, U. S. Naval Engineering Experiment Station.

"Determination of Dissolved Oxygen by Means of the Cambridge Analyzer," by H. A. Grabowski, Combustion Engineering, Inc.

The committee is soliciting papers for a symposium on a phase of radioactivity in water to be presented at the 1958 Annual Meeting of the Society.

The 1957 Max Hecht Award citation and certificate will be presented on June 19, to R. C. Adams.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Issued as a separate publication.

[2] Issued as a separate publication *ASTM STP No. 219.*

The committee consists of 128 members.

There are 71 task groups engaged on projects within subcommittees as follows:

| | |
|---|---|
| Subcommittee I, Advisory | 1 |
| Subcommittee II, Definitions and General Specifications | 5 |
| Subcommittee III, Methods of Sampling and Flow Measurement | 6 |
| Subcommittee IV, Methods of Analysis | 22 |
| Subcommittee V, Methods of Testing | 10 |
| Subcommittee VI, Corrosivity and Performance Testing | 3 |
| Subcommittee VII, Industrial Waste Water | 24 |

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the recommendation for revisions in the Tentative Methods of Test for Sodium and Potassium Ions in Industrial Water and Water-Formed Deposits by Flame Photometry (D 1428 - 56 T).

This recommendation was accepted by the Standards Committee on September 5, 1956, and the revised method appears in the 1956 Supplement to the Book of ASTM Standards, Part 7.

## PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Non-Referee Method of Test for Acidity and Alkalinity in Industrial Water as appended hereto.[3] This method is based on and represents a simplification of the ASTM Method D 1067 - 55 T. It had been requested by the chairman of the Chemical Section, Subcommittee on the Care of Pressure Vessels in Service, of the A.S.M.E. Boiler and Pressure Vessel Committee.

## NEW TENTATIVES

The committee recommends for publication as tentative the following two methods:[4]

Methods of Sampling Homogeneous Industrial Waste Water, and

Method of Test for Iron in High-Purity Industrial Water.

## REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following three methods and continuation of the methods as tentative:

### Tentative Methods of Test for Acidity and Basicity (Alkalinity) in Industrial Water and in Industrial Waste Water (D 1067 - 55 T):[5]

*Section 17.*—Add a new Paragraph (*d*) to read as follows, relettering the subsequent paragraphs accordingly:

> (*d*) *Methyl Purple Indicator* (*pH 5.0*).

*Section 18* (*a*).—Change to read as follows:

> (*a*) Pipet 100 ml of the sample into a 250-ml narrow-mouth Erlenmeyer flask (Note 3). Place the pipet tip at the bottom of the flask while discharging the sample.

*Section 25.*—Change Paragraph (*b*) to read "*Indicator Solutions* (*pH 3.5, 5.0, and 9.0*).—See Section 17 (*c*), (*d*), (*e*), (*f*) and (*h*).

*Section 26* (*a*).—Change to read "Proceed in accordance with Section 18(*a*)."

*Table I.*—Change the second line of the left-hand column to read "pH 5.0 (methyl red or methyl purple) or."

### Tentative Method of Test for Hydrazine in Industrial Water (D 1385 - 55 T):[5]

*Section 2.*—Change the two references to "red" color to read "yellow."

*Section 8* (*b*).—Change the second from last sentence to read, "If it is suspected that oxidation of the hydrazine in the sample is occurring in the interval between collection and analysis, or if the sample is not to be analyzed immediately, collect under acid."

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[3] Appears in "Manual on Industrial Water," December, 1957.

[4] The new and revised tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 7.

[5] 1955 Book of ASTM Standards, Part 7.

*Section 10 (b).*—After the third sentence, add a sentence to read, "Let the mixture stand at least 10 min but not more than 100 min."

**Tentative Method of Test for Nitrite Ion in Industrial Water (D 1254 – 53 T):**[5] Revise as appended hereto.[4]

In Table I are given comparative data for the Rider-Mellon nitrite method as given in Methods D 1254 – 53 T[5] and the proposed revision of Methods D 1254 as appended to this report.[4]

TABLE I.—COMPARATIVE DATA ON NITRITE METHOD.

| Dilutions, ml | $NO_2^-$ Content, ppm | Reading at 545 mμ |
|---|---|---|
| RIDER-MELLON NITRITE METHOD AS IN ASTM METHOD D 1254–53 T[a] | | |
| 0.0 | 0.00 | 0.000 |
| 1.0 | 0.01 | 0.005 |
| 2.0 | 0.02 | 0.013 |
| 4.0 | 0.04 | 0.030 |
| 6.0 | 0.06 | 0.045 |
| 8.0 | 0.08 | 0.060 |
| 10.0 | 0.10 | 0.080 |
| REVISED RIDER-MELLON NITRITE METHOD[b] | | |
| 0.0 | 0.00 | 0.000 |
| 1.0 | 0.01 | 0.003 |
| 2.0 | 0.02 | 0.012 |
| 3.0 | 0.03 | 0.020 |
| 5.0 | 0.05 | 0.040 |
| 6.0 | 0.06 | 0.047 |
| 8.0 | 0.08 | 0.062 |
| 10.0 | 0.10 | 0.085 |

[a] Reagents: Sulfanilic acid in acetic acid and alpha-naphthylamine acetate solution in acetic acid.

[b] Reagents: Sulfanilic acid in concentrated hydrochloric acid and alpha-naphthylamine acetate solution in concentrated hydrochloric acid.

## ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following four tentative methods be approved for reference to letter ballot of the society for adoption as standard without revision:

*Tentative Methods of Test for:*

Total Carbon Dioxide and the Calculation of Bicarbonate and Carbonate Ions in Industrial Water (**D 513 – 54 T**),[5]

Chlorine Requirement of Industrial Water and Industrial Waste Water (D **1291 – 53 T**),[5]

Fluoride Ion in Industrial Water and in Industrial Waste Water (**D 1179 – 55 T**),[5] and

Hardness in Industrial Water (**D 1126 – 55 T**).[5]

## ADOPTION OF TENTATIVES AS STANDARD WITH REVISIONS

The committee recommends that the following two tentative methods be approved for reference to letter ballot of the society for adoption as standard with the revisions as indicated:

**Tentative Methods of Test for Residual Chlorine in Industrial Water (D 1253 – 53 T):**[5]

*Fig. 1.*—Replace the present Fig. 1 with the wiring diagram shown as Fig. 1 of the Tentative Methods of Test for Residual Chlorine in Industrial Waste Water (D 1427 – 56 T).[6]

*Footnote 6.*—Change "Wallace & Tiernan Co., Inc." to read "Wallace & Tiernan, Inc."

*Section 3 (b).*—Change to read as follows:

> (6) Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to the Specifications for Reagent Water (ASTM Designation: D 1193). In addition, the water shall be free of chlorine demand. Prepare the water by adding approximately 20 ppm available chlorine and adjusting the pH to be between 7.0 and 9.0. Protect the prepared water from sunlight for 1 week; then expose it to sunlight until no residual chlorine remains. If desirable, the final dechlorination may be effected with sodium arsenite solution prepared as directed in Section 9 (*c*).

**Tentative Method for Determination of Thickness of Internal Deposits on Tubular Heat Exchange Surfaces (D 1341 – 54 T):**[5]

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[6] 1956 Supplement to Book of ASTM Standards, Part 7.

*Section 6 (a).*—Change to read as follows:

(*a*) Adjust the apparatus for zero by means of brass shims or glazed papers. Select a piece of tubing of the size and weight of those to be surveyed and long enough to accommodate the entire detector head with its carriages. Clean the tubing free of mill scale and thick rust by pickling or wire brushing. Cut brass shim stock or glazed paper, of 0.002-, 0.004-, and 0.006-in thickness, to a size to fit under both contacts of the detector head. Adjust the instrument to the calibrating plate in accordance with instructions. Insert the detector head into the zeroing tube. Make the zero adjustment so that the meter reads slightly up scale from zero (Note 2).

*New Note.*—Add a new Note 2 as follows, renumbering the subsequent notes accordingly:

NOTE 2.—The instrument will not give a reading below the mechanical zero adjustment. Correct readings of shim thickness will not be obtained if the zero adjusting potentiometer is set on the wrong side of the true zero point.

*Section 6 (b).* Change to read as follows:

(*b*) Slip the brass shims or glazed paper singly or in combinations under the contacts of the probe head, depending upon the thickness of scale expected. Compare the instrument reading against the known thickness of the shims or paper. If the reading is heavy, remove the shims or paper and adjust the instrument setting toward zero. If the reading is light, adjust the setting away from zero. Repeat these adjustments until the shim thickness indicated is as nearly correct as possible.

## REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions of the Standard Definitions of Terms Relating to Industrial Water and Industrial Waste Water (**D 1129 - 56**),[6] and accordingly ask for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

*Accuracy.*—The degree of agreement between results of measurement and the true value for the property being measured. The accuracy of a method is expressed quantitatively as the algebraic difference of the average result of a series of controlled determinations from the true value of the property being determined.

NOTE.—Accuracy cannot be stated for a method for which the true value of the property being measured is not known, but a practical estimation can be obtained by comparison of determined results with the generally accepted value.

*Transmittance.*—The ratio of the amount of light ($I$) of known characteristics passing through a column of liquid containing a soluble colorant to the amount of light ($I_o$) from the same source passing through an optically identical column filled with liquid not containing the colorant.

$$T = I/I_o$$

NOTE.—Improved precision and less interference in the measurement of transmittance are obtained as the light used approaches monochromaticity. The use of a ratio for this measurement is desirable because of its freedom from error resulting from transmission losses common to both measurements, effect of absolute intensity of the light source, and partial reflection from the walls of the sample system.

*Absorbance.*—The negative logarithm to the base 10 of transmittance.

$$A = -\log_{10}T$$

NOTE.—This term is often used interchangeably with the older term "optical density."

*Turbidity.*—An optical measure of the concentration of suspended matter in water.

NOTE.—Both relative and absolute measurements of turbidity are employed.

*Jackson Candle Turbidity.*—An empirical measure of turbidity in special apparatus, based on the measurement of the depth of a column of water through which the image of a burning standard candle can just be seen.

*Nephelometric Turbidity.*—An empirical measure of turbidity based on a measurement of the light scattering characteristics (Tyndall effect) of the suspended matter in a sample.

NOTE.—The measurement of nephelometric turbidity is usually accomplished by measuring the intensity of scattered light at approximately 90 deg to the incident beam of light. Numerical values are obtained by comparison with the light-scattering characteristics of a known or arbitrary material in an equivalent optical system or to the transmitted light intensity for a given optical system.

*Absolute Turbidity.*—The fractional decrease of incident monochromatic light through a 1-cm layer of a sample, integrating both scattered and transmitted light.

NOTE.—For the small amount of scattering experienced in essentially colorless solutions, absolute turbidity corresponds to the extinction coefficient in the equation expressing Lambert's law.

*Geometric Light-Scattering Pattern.*—A polar diagram of the intensity of light scattered from a narrow incident beam by a sample in a transparent cylindrical cell.

## TENTATIVES CONTINUED WITHOUT REVISIONS

The committee recommends that all of the tentative methods and specifications under its jurisdiction, with the exception of those recommended for revision or for adoption as standard earlier in this report, be continued as tentative without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I, Advisory* (Max Hecht, chairman).—The over-all activities of the subcommittee included the administration of the work of the committee, methods production, as reported earlier in this report, the preparation of and the approval of the Symposium on Dissolved Oxygen, mentioned earlier in this report, and the consideration of a Symposium for presentation in 1958, relating to Radioactivity in Industrial Water and Industrial Waste Water.

The topical outline of the chapters for a Second Edition of the Manual on Industrial Water was published in the 1956 Report of Committee D-19, indicating the expansion of the chapters to include information on Industrial Waste Water. During the current year, consideration was favorable for the inclusion of an additional chapter on Radioactivity in Industrial Water and Industrial Waste Water.

The committee chairman will advise the Administrative Committee on Research on such matters that may require research study. This contact is in lieu of the proposal of the Research Committee that each of the technical committees of the Society establish either a subcommittee or task group on Research.

J. F. J. Thomas has been appointed to survey the published methods of the committee, to determine variations in concentrations of reagents (where the same reagent is specified in more than one method), and similarly oven drying temperatures.

Committee members elected to serve on the Advisory Subcommittee as members at large for the three-year term ending 1960 are:

A. R. Belyea, Consolidated Edison Company of New York, Inc., and
W. Allan Moore, U. S. Public Health Service, R. A. Taft Sanitary Engineering Center.

Various members of the committee serve as ASTM representatives on committees of other societies. In addition, other members of the committee serve as liaison representatives on other ASTM committees. Each of these committee members filed reports for the information of the committee.

*Subcommittee II on Definitions and General Specifications* (S. K. Love, Chairman).—The Second Edition of the Manual on Industrial Water is being prepared under the direction of this subcommittee. It is expected that the first drafts of all chapters of the new edition will be completed by June 1, 1957 and final drafts by October 1, 1957.

Definitions were approved for accuracy and for several terms in the methods of Test for appearance properties including transmittance, absorbance (optical density), three forms of turbidity, and geometric light-scattering pattern. Definitions for sensitivity and for lowest level of determinability are under consideration.

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee III on Methods of Sampling of Industrial Water* (E. F. Davidson, chairman) is continuing the study of proposed revisions to the Tentative Method of Sampling Steam (D 1066 - 54 T) and Tentative Specifications for Equipment for Sampling Industrial Water and Steam (D 1192 - 51 T) with regard to new methods and equipment which may be required for supercritical operation and monotube system.

Active task groups are working on industrial water to develop the following:

1. Methods of Flow Measurement of Industrial Water.
2. Methods of Sampling Industrial Waste Water.
3. Revision of Chapter VI for the Manual on Industrial Water to cover Sampling and Flow Measurement of Industrial Water.

The task group on sampling has prepared a method for sampling homogeneous industrial waste water for publication as tentative. Work is continuing on a method of sampling waste water containing floating material.

*Subcommittee IV on Methods of Analysis of Industrial Water* (F. E. Clarke, chairman) has completed the new methods for nitrite, low concentration iron (bathophenanthroline method), and routine acidity-basicity determination that are being submitted for publication as tentative. Substantial progress has been made on methods for analysis of deposits, copper, nickel, lead, volatile amines, sulfate and zinc. New projects have been started on methods for micro amounts of chloride, fluoride, organic nitrogen, and for high amounts of nitrite.

*Subcommittee V on Methods of Testing* (A. O. Walker, chairman).—Work on proposed revisions of Methods D 1125 and D 1128 is in progress.

The task group on the flame photometer discussed proposed work on an investigation of gas quality and optimum pressure for operation of particular types of instruments. Section B of Methods D 1428, for high-purity water will be expanded to cover in greater detail the necessary precautions for this analysis.

A final draft of a method of test for hydrogen should be ready for Committee D-19 consideration at the June, 1957, meeting.

The task group on surface tension has completed a draft of a referee method and also the tests to evaluate the precision of the method. A draft of a nonreferee procedure will be prepared, based on the Traube Stalagomometer.

The Task Group on Measurement of Radioactivity held its first meeting at Charleston. The group proposed that, rather than develop a method for sodium-24 and potassium-42, a method be prepared for gamma-emitting isotopes having gamma energies above 0.5 Mev. This would be usable for sodium and potassium, as well as quite a number of other possible tracer isotopes.

The task groups on viscosity and on settling velocities were dropped for lack of interest.

*Subcommittee VI on Corrosivity and Performance Testing* (F. N. Alquist, chairman).—The Task Group on Service Testing of Tubular Metallic Products has in the past two meetings reduced to two problems the unfinished tasks on writing up this method. It is expected to be complete at the next meeting.

The Task Group on ion-exchange materials is making progress on preparing methods of performance testing of cation exchange materials.

The subcommittee is making a study of all past work on water which is radioactive in order to prepare a chapter for the Second Edition of the Manual of Industrial Water. In addition, a symposium is being planned for the 1958 Annual Meeting. The papers of this symposium will discuss the measurement of radioactivity in water, the handling of industrial waste water for atomic power production, the disposal of water waste from use of radioisotopes, and underground disposal of water from nuclear operations.

*Subcommittee VII on Industrial Waste Water* (L. D. Betz, chairman).—At the present time there are 16 active task groups working on this subcommittee, one less than those existing at the time of the last report, the task group on Organic Nitrogen having been transferred to Subcommittee IV. Several of the task groups of Subcommittee VII are working jointly with task groups of Subcommittee IV with a view of developing methods that will be applicable to the analyses of both industrial water and waste water.

Subcommittee VII, acting in conjunction with Subcommittee IV, has proposed a tentative method on Acidity and Basicity.

The task group on detergents has changed its name to Synthetic Surface-Active Materials. These components are further defined as synthetic materials that at use concentration show surface activity in the form of surface tension lowering, foaming, or surface adsorption. Definitions for anionic, cationic, and nonionic synthetic surface active materials were established. The work of the task group will first be limited to a method for determining anionic surface active materials. An extensive literature search was prepared as a preliminary step.

The Task Group on Oily Matter is evaluating the precision and accuracy of the method of test for chloroform-extractable matter.

The Task Group on Oxygen Demand approved investigation into a minor change in the method for chemical oxygen demand. Work on a method for dissolved oxygen is expected to result shortly in recommendation for acceptance as a tentative. When the method for dissolved oxygen is finalized, the method for B.O.D. will proceed.

The Task Group on Specific Gravity is investigating the possibility of using the gravitometer as a nonreferee procedure.

The Task Group on Sulfur Compounds is investigating the substitution of para-aminodimethyl aniline sulfate as a substitute for distilled amine in Method D 1255 - T.

The Task Group on Phenols is undertaking round-robin tests at the 100 ppb level after successful completion of work at lower phenol levels.

The Task Group on Taste and Odor is undertaking a comparison of Method D 1292 with another procedure to determine if substitution or presentation of both methods will be recommended.

The Task Group on Chromium is studying a persulfate - permanganate method to replace the permanganate-azide method. A hypobromite modification will be retained and cooperative testing will be undertaken.

The Task Group on Cyanides has evolved a method which will permit two choices of completion depending on cyanide concentration. Both the titration and colorimetric variations are under study.

A non-referee method for acidity and basicity is under study. Other methods are being developed by task groups on toxicity to aquatic life, reporting analytical results, gaging, and chlorine residual.

This report has been submitted to letter ballot of the committee, which consists of 128 members; 83 members have returned their ballots, of whom 81 voted affirmatively and 00 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
*Chairman.*

O. M. ELLIOTT,
*Secretary.*

### EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the following recommendations:

*New Tentative Method of Test for:*

Oxidation-Reduction Potential of Industrial Water (D 1498 - 57 T)

*Revision of Tentative Method of Test for:*

Iron in High Purity Water (D 1497 - 57 T)

These recommendations were accepted by the Standards Committee on September 13, 1957, and the new and revised tentative methods appear in the 1957 Supplement to Book of ASTM Standards, Part 7.

# REPORT OF COMMITTEE D-20

## ON

## PLASTICS*

Committee D-20 on Plastics held three meetings during the year: at Atlantic City, N. J., on June 18 to 20, 1956; at St. Louis, Mo., on October 15 to 17, 1956; and at Roanoke, Va., on February 27 to March 1, 1957.

The committee consists of 518 members, of whom 188 are classified as producers, 138 as consumers, and 192 as general interest members. There are 210 voting members who were designated by their companies as a result of a recent questionnaire to bring the records up to date. Ballots are sent only to voting members.

The committee has systematized procedures for consideration of new member applications. Application forms are proving helpful in providing information on new applications to appropriate officers of the committee.

The United States participates in the work of Technical Committee 61 of the International Organization for Standardization (ISO), through its American Group, C. H. Adams, Leader. Representatives from the United States attended the sixth meeting of ISO/TC 61 at The Hague, The Netherlands, on September 17 to 22, 1956. This group made outstanding progress in developing international standard methods of test. A report of the meeting at The Hague appeared in the December 1956 issue of the ASTM BULLETIN.

The ISO Committee 61 on Plastics accepted the invitation of the American Delegation to hold the 1958 meeting in this country. It is planned to hold the Committee D-20 meeting and a symposium on plastics testing and standards in Philadelphia, Pa. on October 28 to 31, 1958, and the ISO meetings in Washington, D. C., on November 3 to 8, 1958.

The committee has agreed by letter ballot to recommend to the Society as an editorial policy the use of certain nomenclature on high polymers approved by the International Union of Pure and Applied Chemistry (IUPAC). For example, the term poly(vinyl chloride) as used in this report conforms to the recommendation.

An orientation course to indoctrinate new officers of sections and subcomittees and to make the operations more uniform and efficient was initiated. This course will consist of a series of one hour discussions at each spring and fall meeting.

The offices of Meeting Secretary and Financial Secretary were created to reduce the work load on the General Secretary. B. L. Lewis was appointed Meeting Secretary and Frank Y. Speight, Financial Secretary.

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957

*New Tentative Specification for:*

Tetrafluoroethylene Resin Molding and Extrusion Materials (D 1457 – 56 T)

*Revision of Tentative Methods of Test for:*

Tensile Properties of Thin Plastic Sheets and Films (D 882 – 54 T)
Tensile Properties of Plastics (D 638 – 52 T)

These recommendations were accepted by the Administrative Committee on December 17, 1957, and they appear in the February, 1957, Compilation of ASTM Standards on Plastics.

## RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication six new tentatives, and is recommending the revision of tentatives and the adoption as standard of tentatives. The new tentatives, the standards affected, together with the revisions recommended are given in detail in the Appendix.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Mechanical Properties* (C. H. Adams, chairman) prepared the revisions of the Tentative Methods of Test for Tensile Properties of Plastics (D 638 – 56 T) and the Tensile Properties of Thin Plastic Sheets and Films (D 882 – 56 T) referred to above. Work is in progress on a tension test method based on the use of small specimens. The tear strength method (D 1004 – 49 T) will be revised to reflect the recent improvements made in tensile strength methods D 882 on which it depends. A proposed method for impact testing of plastic sheeting is under consideration. A round-robin project on tensile-impact-toughness is nearing completion. Problems in the area of thin laminates have slowed the revision of Tentative Method of Test for Flexural Properties of Plastics (D 790 – 49 T). The proposed tensile-modulus method for thin films is essentially completed. A method for scratch hardness based on the Bierbaum technique is being investigated. Development of a blocking method has been delayed by unexpected technical problems. Joint jurisdiction has been requested with Committee D-11 on the method for durometer hardness of rubber (D 676 – 55 T). Revisions will be proposed to adapt the method for testing plastics as well as rubber.

*Subcommittee II on Effects of Radiation* (D. S. Ballantine, chairman) is a joint subcommittee with Committee D-9 on Electrical Insulating Materials, and the organization meeting was held on February 28, 1957. Initial organization comprises Sections A on Nomenclature and Definitions, B on Dosimetry, C on Correlation of Radiation Sources, D on Effects of Total Dosages and Dose Rate, and E on Post Irradiation Effects. Due to considerable overlapping or interrelation of interests among the sections, some reorganization is indicated.

It is expected that initial consideration will be confined to organic materials which are more susceptible to radiation damage than inorganic materials. This will greatly simplify the problems of dosimetry.

*Subcommittee III on Thermal Properties* (R. M. Berg, chairman).—Work on evaluation of the ZST (zero-strength-time) test for polyethylene characterization has proceeded to the point where tentative test conditions have been established and a round-robin almost finished. The section on flammability has been studying tests for ignition properties of plastic materials, flammability of plastic foam, and a radiant-panel flame-spread test. The group working on procedures for flow of thermoplastics completed a revision of Method D 1238 and is currently drafting a new method for flow rates at high rates of shear. A round robin is in process to evaluate the SPI test for low-temperature brittleness of plastic films. A new Section J was formed to develop a softening point test for polyethylene using the Vicat apparatus.

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[1] See p. 410.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee IV on Optical Properties* (W. E. Brown, chairman) is collecting data on clarity of films, yellowness determinations, and hiding power of diffusing plastics by several methods. Transparency by various instrumental methods, haze determined by the method for Haze and Luminous Transmittance of Transparent Plastics (D 1003 – 52) and visual ranking of films covering a wide clarity range shows that further differentiation among the terms transparency, haze, and gloss is needed. Methods for yellowness based on spectrophotometric and related equipment are being studied. A new section on hiding power (image obscuration) is conducting interlaboratory tests on light-diffusing plastics in conjunction with needs of the joint SPI-IES-NEMA Subcommittee on Plastics in Lighting. Narrow angle scattering shows promise in both clarity and hiding power measurements. The Method of Test for Light Transmission of Corrugated Plastic Panels, developed by Subcommittee XVIII and the Society of Plastics Industry, is being reviewed.

*Subcommittee V on Permanence Properties* (B. G. Achhammer, chairman) has developed an acrylic plastic base containing a yellow dye which will be evaluated as a fading standard to determine equivalent amounts of exposure to ultraviolet radiant energy in artificial weathering machines. A test method for artificial weathering of plastics by carbon-arc machines is being written based on Committee E-1's recommended practice for operation of such equipment. The Method of Test for Water Absorption of Plastics (D 570 – 54 T) is being revised to include a long-time water absorption test. A recommended practice for use of fluorescent sunlamps in artificial weathering is being developed. A new Section J was formed to develop test methods and specifications for the corrosivity of plastics.

*Subcommittee VII on General Analytical Methods* (M. R. Rector, chairman) prepared the proposed Method of Test for the Measurement of Density of Plastics by the Density-Gradient Technique as appended to this report.[3]

Round-robin tests on carbon black dispersion in polyethylene and on dilute-solution viscosities of vinyl resins yielded data which indicates these methods may soon be submitted to appropriate ballot. A particle size round-robin will be conducted before drafting a method.

A paper was presented by R. M. Schulken, Jr., on the determination of absorptivity of black polyethylene films using a photo-multiplier type photometer.

*Subcommittee VIII on Research* (A. G. H. Dietz, chairman) has continued to arrange for presentation of papers on subjects of current interest to the committee.

At the fall meeting four papers were presented:

"Thermal Stability Developments in Committee D-9" by K. N. Mathes of the General Electric Co.

"The Testing of Poly(Vinyl Chloride) Electrical Formulations" by G. W. Ashworth, J. R. Darby, W. E. Koerner and R. H. Munch of the Monsanto Chemical Co.

"A New Test Method for the Evaluation of Chemical Finishes on Glass Roving" by P. W. Erickson and I. Silver of the U. S. Naval Ordnance Laboratory.

"An Instrument for Measurement of Clarity of Polyethylene Film" by R. J. Clark, H. C. Tucker and R. F. Wall of the Monsanto Chemical Co.

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[3] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 6.

At the spring meeting the program was as follows:

"Measurement of the Elastic Modulus of Films" by J. K. Owens and G. W. Strain of E. I. du Pont de Nemours & Co.

"Use of Time Temperature Curves in Characterizing Polyethylene" by J. L. Silver of the Bakelite Co.

Round-table Discussion of Development of Working Stresses. R. H. Carey, A. G. H. Dietz, H. A. Perry, H. W. Kuhlman, and F. W. Reinhart participated in the discussion.

At the request of the Administrative Committee on Research, the committee has distributed a questionnaire toward assembling a list of plastics research problems to contribute to the ACR project "Challenges in Materials Research."

*Subcommittee IX on Molds and Molding* (G. H. Williams, Jr., chairman) is working toward establishing standard molding procedures for phenolic plastics as well as transfer dies and test-specimen standards. Another active project is the development of an injection molding procedure for Specification D 703 Type 3 polystyrene. The effect of lubricants on the molding conditions of plastic materials is being investigated.

The subcommittee has been reorganized, abolishing previous sections and establishing new sections A through I on Amino Test Specimens, Phenolic Test Specimens, Shrinkage, Transfer Dies for Test Specimens, Mold Temperature and Pressure, Injection Processes, and Sheet Preparation.

*Subcommittee X on Definitions, Nomenclature and Significance of Tests* (P. E. Willard, chairman) has remained active in preparing definitions of technical terms used in the plastics industry. Definitions for 10 terms were approved during the year and have been added as a revision of Definition D 883 as appended to this report.

A program to establish standard abbreviations for plastics terms has been initiated.

Definitions for 36 additional terms are in various stages of preparation.

The subcommittee plans to review definitions and equivalent terms prepared by ISO/TC61 on Plastics.

*Subcommittee XIV on Conditioning* (G. M. Armstrong, chairman) is considering suggestions to revise the Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 54) to include a system of descriptive nomenclature similar to that used in MIL-P specifications for specifying conditioning procedures and to clarify the significance of tolerances for humidity and temperature. The subcommittee is cooperating in development of international conditioning standards in committees of the International Organization for Standardization and the International Electrochemical Commission. The work of this subcommittee is a joint activity with Committee D-9 on Electrical Insulating Materials.

*Subcommittee XV on Thermoplastic Materials* (A. C. Webber, chairman) prepared the new Tentative Specification for Polytetrafluoroethylene Molding and Extrusion Materials (D 1457 - 56 T), and revisions of Specifications D 703 and D 788 covering polystyrene and methacrylate molding and extrusion compounds.

Work is in progress on new specifications for cellulose propionate, rigid poly(vinyl chloride), and styrene-rubber molding materials and polytetrafluoroethylene rods. A new Section N is developing a specification for acrylonitrile-butadiene-styrene (ABS) plastics. A revision of the specifications for nylon molding and extrusion materials is about complete.

High in importance on the subcommittee agenda has been the work of Section J towards the preparation of a badly needed revision of the Specification for Polyethylene Molding and Extrusion Materials, D 1248. Five task groups have been actively developing methods needed for this revision.

*Subcommittee XVI on Thermosetting Materials* (G. D. Johnston, chairman) has completed a comprehensive revision of the phenolic specification D 700 to bring it up to date, and is preparing a revision of the electrical requirements of urea-formaldehyde plastics types 1 and 2 of specification D 705, and the addition of a new high impact grade of polyester to specification D 1201.

A new allyl plastic specification and a revision of the melamine plastic specification, D 704, will be ready soon.

Interest is being solicited toward establishing a section on casting compounds.

*Subcommittee XVII on Plastic Pipe and Fittings* (G. H. Reed, chairman) consists of over 100 members with six sections working on dimensional and performance requirements and test methods for rigid poly(vinyl chloride), acrylonitrile - butadiene - styrene copolymers, polyethylene, and cellulose acetate butyrate pipe and pipe fittings.

The subcommittee prepared the proposed specification for butyrate pipe as appended to this report. Projects are in progress to develop standards for PVC pipe, polyethylene pipe, and ABS pipe. Standards for fittings and test methods for creep rupture are also being developed.

*Subcommittee XVIII on Reinforced Plastics* (R. B. Crepps, chairman) established a new Section G on High-Temperature Resistant Materials. Section activity was continued on reinforced pipe, corrugated panels, and laminating resins.

The subcommittee, in cooperation with the Society of Plastics Industry, prepared the proposed tentative method for transverse strength of corrugated panels appended to this report.[3]

*Subcommittee XIX on Plastic Film and Sheeting* (T. D. Mecca, chairman) is preparing final drafts of specifications for non-rigid poly(vinyl chloride) plastic film and for biaxially oriented polystyrene plastic sheet. Work is also in progress on specifications for rigid vinyl sheeting. Specifications are being drafted for a general purpose polyethylene film in close liaison with the Society of the Plastics Industry. Editorial changes and a round-robin study to define necessary changes in test conditions are being made in connection with a proposed specification for extruded acrylic plastic sheet. The subcommittee prepared the proposed Tentative Method of Test for Orientation Stress of Plastic Sheeting appended to this report. Action has been initiated on the preparation of specifications for styrene-rubber and acrylonitrile-butadiene-styrene plastic sheet.

*Subcommittee XX on Cellular Plastics* (I. T. Stoneback, chairman).—Following the June meeting, the membership was canvassed to ascertain fields of interest. Based on the response the subcommittee organized sections A through E covering test methods, and urethane, poly(vinyl chloride), polyethylene, and polystyrene cellular plastics.

This report has been submitted to letter ballot of the committee, which consists of 210 voting members; 122 members returned their ballots, of whom 108 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. W. REINHART,
*Chairman.*

J. B. DECOSTE,
*Secretary.*

## EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards a new Tentative Method of Test for Diffuse Light Transmission Factor of Reinforced Plastics Panels. The new tentative method was accepted by the Standards Committee on December 13, 1957, and it is available as a separate reprint bearing the designation D 1494 — 57 T.

## APPENDIX

## RECOMMENDATIONS AFFECTING STANDARDS ON PLASTICS

In this Appendix are listed six new tentatives and proposed revisions in certain standards and tentatives which are referred to earlier in this report. These standards and tentatives appear in their present form in the 1955 Book of ASTM Standards, Part 6, or in the 1956 Supplement to Book of ASTM Standards, Part 6.

### NEW TENTATIVES

The committee recommends for publication as tentative the following as appended hereto:[1]

### Recommended Practice for Resistance of Plastics to Artificial Weathering Using Fluorescent Light and Fog Chamber

This aging test is designed to simulate outdoor exposure conditions on a laboratory scale.

### Method of Test for Transverse Strength of Corrugated Reinforced Plastic Panels

This method covers a test for determining the ultimate transverse load of corrugated reinforced translucent building panels.

### Specifications for Extruded Cellulose Acetate Butyrate Pipe

Specifications are needed to control the quality of this pipe which has become a regular article of commerce. These specifications cover the requirements, dimensions, and tolerances of pipe extruded from cellulose acetate butyrate materials.

### Method of Test for Orientation Release Stress of Plastic Sheeting

Orientation stress or internal locked stress from the manufacturing process influences physical properties and therefore can be a useful criterion for specification purposes.

### Method of Test for the Measurement of the Density of Plastics by the Density-Gradient Technique

This method provides a means for rapidly determining the density of a plastic to a greater accuracy than has been heretofore possible. The method is designed to yield results accurate to better than 0.05 per cent.

### REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives:

### Tentative Method of Test for Brittleness Temperature of Plastics and Elastomers by Impact (D 746 – 55 T):

*Section 10.*—Add a new Paragraph (*4*) to read, "Type of apparatus used," and renumber the present Paragraph (*4*) and subsequent paragraphs accordingly.

*Section 4*(*a*).—Add as the second sentence, "Each individual specimen shall be firmly and securely held in the clamp."

*Section 5.*—Add the following note to this section:

NOTE.—Sharp dies must be used in the preparation of specimens for this test if reliable results are to be achieved. Careful maintenance of die cutting edges is of extreme importance and can be obtained by daily lightly honing and touching up the cutting edges with jewelers' hard Arkansas honing stones. The condition of the die may be judged by investigating the rupture point on any series of broken specimens. When broken specimens are removed from the clamps of the testing machine it is advantageous to pile these specimens and note if there is any tendency to break at or near the same portion of each specimen. Rupture points consistently at the same place may be the indication that the die is dull, nicked, or bent at that particular position.

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[1] The new and revised tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 6.

### Tentative Method of Test for Water Absorption of Plastics (D 570 – 54 T):

This method has been extensively revised and is appended hereto in its proposed revised form.[1]

### Tentative Definitions of Terms Relating to Plastics (D 883 – 56 T):

Add the following new definitions:

*Compression Molding.*—A method of forming objects from plastics by applying pressure and usually heat to the material contained in a confining mold cavity.

*Gamma Transition, Glassy Transition.*—The change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one.

NOTE.—This transition generally occurs over a relatively narrow temperature region and is similar to the solidification of a liquid to a glassy state; it is not a phase transition. Not only do hardness and brittleness undergo rapid changes in this temperature region but other properties such as thermal expansibility and specific heat also change rapidly. This phenomenon has been called second order transition, glass transition, rubber transition, and rubbery transition. The word transformation has also been used instead of transition.

*Gamma Transition Temperature, Glassy Transition Temperature.*—The temperature region in which the gamma or glassy transition occurs. (See Gamma or Glassy Transition.)

NOTE.—The measured value of gamma or glassy transition temperature depends to some extent on the details of the method of test.

*Glass.*—An inorganic product of fusion which has cooled to a rigid condition without crystallizing.

NOTE.—Glass is typically hard and brittle and has a conchoidal fracture. (See Standard Definitions of Terms Relating to Glass and Glass Products (ASTM Designation: C 162 – 56).

*Hydrocarbon Plastics, n.*—Plastics based on resins composed of carbon and hydrogen alone.

*Isocyanate Plastics, n.*—Plastics based on resins made by the condensation of organic isocyanates with other compounds.

*Polyethylene, n.*—A plastic or resin prepared by the polymerization of ethylene as essentially the sole monomer.

*Polypropylene, n.*—A plastic or resin prepared by the polymerization of propylene as essentially the sole monomer.

*Postforming, n.*—A method of shaping substantially cured thermoset plastic sheets or other forms by heating and stressing into other configurations.

*Syneresis, n.*—The contraction of a gel.

NOTE.—This is usually evidenced by the separation of liquid. (See first definition of Gel.)

Delete the definition of stress-crack. This definition is being transferred to the Tentative Nomenclature D 675 – 55 T.

### Tentative Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 – 55 T):

Add the definition of stress-crack appearing in the Tentative Definitions of Terms Relating to Plastics (D 883 – 55 T).

Revise the definitions for mottle, and plate-mark to read as follows:

*Mottle.*—An irregular distribution or mixture of colorants or colored materials giving a more or less distinct appearance of specks, spots, or streaks of color.

NOTE.—Mottling is often purposely achieved although it may occur accidentally due to improper mixing.

*Plate-Mark.*—An imperfection in a pressed plastic sheet resulting from the surface of the pressing plate.

### Tentative Specifications for Phenolic Molding Compounds (D 700 – 55 T):

Revise as appended hereto.[1] The revisions bring the requirements into agreement with current commercial materials and the elimination of dependence on Methods D 48.

**Tentative Method of Test for Measuring Flow Rate of Thermoplastics by Extrusion Plastometer (D 1238 – 52 T):**

Revise as appended hereto.[1] In its original form the test was primarily intended for polyethylene. The changes recommended in the test conditions of temperature and load broaden its application to other polymers.

### REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

**Standard Method of Test for the Colorfastness of Plastics to Light (D 620 – 49):**

*Section 2(a).*—Change "550" to read "300" hr.

*Section 4.*—In the second sentence change "bottom" to read "top" plane.

**Standard Recommended Practice for Accelerated Weathering of Plastics Using S-1 Bulb and Fog Chamber (D 795 – 49):**

*Section 2(a).*—At the end of the first sentence, change "550" to read "300" hr.

### ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Testing Cellulose Acetate Butyrate (**D 817 – 55 T**) be approved for reference to letter ballot of the Society for adoption as standard with the following revisions:

*New Section.*—Add a new Section 7 as follows, renumbering the present Section 7 and subsequent sections accordingly:

7. *Choice of Method.*—The following Methods A and B may not give identical results on the same sample. The method to be used should be agreed upon by the supplier and purchaser.

*Section 8(b).*—Renumber as Section 9(*b*) and change "in extracting" to read "for."

*Section 25(f).*—Renumber as Section 26(*f*) and in the eighth line change "1 min" to read "2 min."

*Section 28(a).*—Renumber as Section 29(*a*) and in the tenth line change "1 min" to read "2 min."

### ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

*Tentative Method of Test for:*
Bursting Strength of Round Rigid Plastic Tubing (**D 1180 – 51 T**), and
Measuring the Molding Index of Thermosetting Molding Powder (**D 731 – 55 T**).
*Tentative Specifications for:*
Molds for Test Specimens of Plastic Molding Materials (**D 647 – 54 T**), and
Vinylidene Chloride Molding Compounds (**D 729 – 52 T**).

### REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

**Standard Method of Test for Ammonia in Phenol-Formaldehyde Molded Materials (D 834 – 49):**

*Section 1.*—Delete the words "and molding powders."

# REPORT OF COMMITTEE D-21
## ON
# WAX POLISHES AND RELATED MATERIALS*

Committee D-21 on Wax Polishes and Related Materials held one meeting during the year: in Washington, D. C., on December 6, 1956.

Additions and changes during the year brought the membership of the committee to 69; of whom 32 are classified as producers, 14 as consumers, 23 as general interest members, and one consulting member.

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee D-21 presented to the Society, through the Administrative Committee on Standards, the proposed Tentative Method of Test for 60 deg Specular Gloss of Emulsion Floor Polish (D 1455 - 56 T). This recommendation was accepted by the Standards Committee on September 5, 1956, and the new method appears in the 1956 Supplement to Book of ASTM Standards, Part 4.

### ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

Committee D-21 recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

*Tentative Method of Test for:*

Total Ash and Silica in Water-Emulsion Waxes (**D 1288 - 53 T**),[1]

Sediment in Water-Emulsion Waxes by Centrifuge (**D 1290 - 53 T**),[1] and

Paraffin-Type Hydrocarbons in Carnauba Wax (**D 1342 - 56 T**).[2]

### TENTATIVES CONTINUED WITHOUT REVISION

The subcommittees of Committee D-21 have reviewed existing methods of test which have been published by the Society for two years or more and the committee recommends that the following be continued as tentative without revision:

*Tentative Method of Test for:*

Nonvolatile Matter (Total Solids) of Water Emulsion Waxes (**D 1289 - 55 T**),

Acid Number (Empirical) of Natural Waxes (**D 1386 - 55 T**), and

Saponification Number (Empirical) of Natural Waxes (**D 1387 - 55 T**).

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Nomenclature* (J. T. Hohnstine, chairman) is preparing a glossary of terms relating to wax polishes.

*Subcommittee II on Raw Materials* (Melvin Fuld, chairman) is working on revisions of Tentative Methods D 1386 - 55 T and D 1387 - 55 T for the acid number and saponification number of waxes. Task groups are preparing additional test methods for various properties of natural waxes.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 4.

[2] 1956 Supplement to Book of ASTM Standards, Part 4.

[3] The letter ballot vote on these recomendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee III on Physical and Chemical Testing* (Gerald DeNapoli, chairman) has work under way on a test method for the stability of water emulsion waxes, and a method for the determination of nonvolatile matter in solvent waxes.

A task group is investigating methods for the determination of silicones in wax polishes.

*Subcommittee IV on Performance Tests* (C. S. Kimball, chairman) is working on methods for testing the water spotting and removability of floor waxes. A task group is also investigating methods of determining the soil resistance of water emulsion waxes. Work is progressing on a report on the significance of slip tests on floors by the James and Sigler machines.

*Subcommittee V on Specifications* (Wm. Joy, chairman) is preparing a specification for industrial type water emulsion floor waxes. Completion of this work awaits the development by the other subcommittees of test methods for several of the properties involved.

This report has been submitted to letter ballot of the committee, which consists of 69 members; 43 members have returned their ballots, of whom 37 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. W. WALTON,
*Chairman.*

B. S. JOHNSON,
*Secretary.*

# REPORT OF COMMITTEE D-22
ON
# METHODS OF ATMOSPHERIC SAMPLING AND ANALYSIS*

Committee D-22 on Methods of Atmospheric Sampling and Analysis held two meetings during the year: on September 18, 1956, in Atlantic City N. J., in connection with the Air Pollution Symposium of the American Chemical Society, and on February 5, 1957, in Philadelphia, Pa.

## Adoption of Tentative as Standard With Revision

The committee recommends that the Tentative Method for Determining Odor Concentration in Air and Gases (Dilution Method) (**D 1391 – 56 T**)[1] be approved for reference to letter ballot of the Society for Adoption as standard with the following change:

*Title.*—Change the title to read, "Method of Measurement of Odor in Atmospheres (Dilution Method)."

## Adoption of Tentative as Standard Without Revision

The committee recommends that the Tentative Recommended Practice for Planning the Sampling of the Atmosphere for Analysis (**D 1357 – 55 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## Activities of Subcommittees

*Subcommittee I on Nomenclature and Units* (F. A. Patty, chairman) has completed 30 additional definitions which, upon approval by the committee, are to be added to Definitions D 1356. The former chairman of the Task Group on Units, Kenneth Morse, had completed a list of units on which conversion factors are desired. Paul Halley, new chairman of this task group, is preparing a report on conversion factors for these units. The subcommittee agreed to use conversion factors only to five significant figures even though a greater number of significant figures is available for a given conversion factor. Exception would be made for conversion between cubic centimeter and milliliter. The Task Group on Symbols, of which Fred Mallette is chairman, is preparing a report on this subject.

*Subcommittee II on Sampling* (Leslie Silverman, chairman) recommended the adoption as standard of Tentative Recommended Practice D 1357, referred to earlier in the report.

Sampling methods for gases and vapors will be referred to committee ballot after several suggested changes have been incorporated. As part of the work on sampling particulate matter, a chart showing application of the several methods to various situations is being prepared by W. J. Smith as a modification of the Air Pollution Control Assn. material to meet ASTM requirements. A number of separate methods will be prepared, each to discuss one of the classifications of sampling as settling jars, filter devices, and impingers.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 7.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Methods of sampling for particle size determination have been essentially completed by M. W. First and after editing will be referred to ballot of the committee.

Work is also under way on stack sampling and sampling of vegetation, soils, and miscellaneous matter.

*Subcommittee III on Analytical Methods* (J. Cholak, chairman) has completed a proposed tentative method for determination of fluorides in the atmosphere. Work is in process on methods of analysis of fluorides in vegetation and biological material.

For the determination of nitrogen dioxide, there is now being prepared a draft of a phenoldisulfonic acid method for collection of grab samples in stacks where concentrations in excess of 50 ppm are contemplated.

In the development of a method for total sulfate, it was found that nitrogen dioxide may interfere at certain levels but not at the low concentrations existing in the atmosphere.

All of the three oxidant determination methods investigated have been found in good agreement provided the impinger is used as the sampling device rather than the fritted glass dispersion unit.

*Subcommittee IV on Instrumentation* (M. D. Thomas, chairman) is preparing a method for determination of particulate matter based on optical density of the filtered material. The Gucker-O'Konski method is to be drafted in ASTM form, and since results from this right-angle light-scattering method do not vary significantly from those of the Sinclair small-angle forward light-scattering method, these two will be included as parts A and B in the same report. It was suggested that the two methods be reported separately, as the Sinclair method gives data which can be converted to the mass of the particulate matter whereas the Gucker-O'Konski method gives the number of particles per liter with a particle size analysis on dynamic samples.

Reports on a directional particulate-fall apparatus and oxidant determination methods have been prepared and are being reviewed editorially.

The method for nitrogen oxides as described in a paper in the December 1956 issue of *Analytical Chemistry* appears to be suitable as an ASTM method and a draft will be prepared.

Respectfully submitted on behalf of the committee,

L. C. McCABE,
*Chairman.*

W. A. COOK,
*Secretary.*

# REPORT OF COMMITTEE D-23

ON

# CELLULOSE AND CELLULOSE DERIVATIVES*

Committee D-23 on Cellulose and Cellulose Derivatives held one meeting during the year: in Atlantic City, N. J., on September 19, 1956. On September 20 a joint meeting was held with the International Committee for Cellulose Analysis (ICCA). The Executive Subcommittee met also in New York City on February 22, 1956.

The committee consists of 46 members of whom 14 are classified as producers, 11 as consumers, and 21 as general interest members.

## ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Testing Cellulose Acetate Butyrate (**D 817 - 55 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard with the following revision:

*New Section.*—Under the heading Free Acidity, add a new Section 7 as follows, renumbering the subsequent sections accordingly:

> 7. *Choice of Method.*—The following Methods A and B may not give identical results on the same sample. The method to be used should be agreed upon by the supplier and purchaser.

*Section 8(b).*—Renumber as Section 9(b), and change "in extracting" to read "for."

*Section 25(f).*—Renumber as Section 26(*f*), and in the eighth line change "1 min" to read "2 min."

*Section 28(a).*—Renumber as Section 29(*a*), and in the tenth line change "1 min" to read "2 min."

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Nomenclature and Specifications* (Kyle Ward, Jr., chairman) is preparing definitions for 25 terms.

*Subcommittee II on Cellulose* (O. A. Battista, chairman) is assembling seven bales of standard wood pulps and linters at the Memphis, Tenn., plant of the Buckeye Cellulose Corp. These are to be used as standard samples in connection with interlaboratory testing of the various methods of cellulose analyses under consideration by the committee. They will also be used for international evaluation of these and similar cellulose methods by the ICCA. Storage of duplicates of some or all of the standard cellulose samples at Uddeholm in Sweden, and distribution to interested persons on the Continent by Miss Karin Wilson, Chairman of ICCA, is being considered.

Availability of standard samples should expedite the work of the various sections toward establishing as standard a number of cellulose methods that have been under consideration for some time. The following procedures will be evaluated: (*1*) Ash (*2*) Non-dilution alkali solubility, (3) Disperse viscosity, (4) Whiteness, (5) Chain length distribution, and (6) Moisture by Karl Fischer Method.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1955 Book of ASTM Standards, Part 4.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

These samples also will be used to compare the ASTM aniline acetate method for pentosans (D 1438 - 56 T) with the TAPPI bromination procedure and the Swedish orcinol procedure. It is hoped that the new sections for methods of test for functional groups, as well as the application of chromatographic procedures for non-cellulosic components, may also be activated with the availability of these standard samples.

*Subcommittee III on Organic Esters* (L. B. Genung, chairman) recommended the adoption as standard of the Tentative Methods of Testing Cellulose Acetate Butyrate (D 817 - 55 T). Test methods under consideration include measurement of hydroxyl groups and de-esterification of cellulose esters for determination of viscosity of cellulose.

*Subcommittee V on Cellulose Ethers* (R. W. Swinehart, chairman) is continuing work on an assay procedure for determining the purity of sodium carboxymethylcellulose. The hot-leach method has been selected for final interlaboratory testing as soon as a statistically planned experiment is designed.

*Subcommittee VI on Statistics* (A. F. Johnson, chairman) has completed sections of the statistics manual. Requests for assistance on planning of experiments have been received and handled on interlaboratory testing of the Karl Fischer moisture method and comparison of caustic extraction procedures.

This report has been submitted to letter ballot of the committee, which consists of 46 members; 29 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

O. A. BATTISTA,
*Vice-Chairman.*

W. W. BECKER,
*Secretary.*

# REPORT OF COMMITTEE D-24
ON
# CARBON BLACK*

Committee D-24 on Carbon Black held two meetings during the year: the organization meeting on June 22, 1956, in Atlantic City, N. J., and in Philadelphia, Pa., on February 6 and 7, 1957, during ASTM Committee Week.

The formation of Committee D-24 was approved at the September 20, 1955, meeting of the ASTM Board of Directors, in response to a request by a group of representatives of carbon black producers who had informally organized as the Carbon Black Industry Committee. They had subjected a number of selected applicable test methods to cooperative testing to determine their reproducibility toward establishing suitable standard test methods.

The officers elected at the organization meeting for the ensuing term of two years are as follows:

Chairman, S. R. Doner.
Vice-Chairman, C. A. Carlton.
Secretary, A. G. Cobbe.

Thirteen test methods developed by the Carbon Black Industry Committee were published in the July 1956 issue of the ASTM BULLETIN as information only. These methods formed the basis for development of the proposed tentative methods of test appended to this report.

The committee consists of 41 members; of whom 11 are classified as producers, 24 as consumers, and 6 as general interest members.

## NEW TENTATIVES

The committee recommends for publication as tentative the following methods of test as appended hereto.[1]

### Method of Test for Pellet Size Distribution of Carbon Black:

This method describes the procedure for separating a pelleted carbon black sample into six portions depending upon the size of the pellets.

### Method of Test for Attrition of Pelleted Carbon Black:

This method describes a procedure for determining the amount of breakdown of carbon black pellets which are subjected to severe mechanical work. Good mechanical stability is required of carbon blacks which are to be used in a bulk-handling system.

### Method of Test for Sieve Residue from Carbon Black:

This method describes a procedure for determining the amount of coke-like nondispersible material which may be present in carbon black.

### Method of Test for Pour Density of Pelleted Carbon Black:

The pour density of carbon black is related to the amount of pelleted black than can be stored in a given volume.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 6.

**Method of Test for Fines Content of Pelleted Carbon Black:**

Fines or dust are important in the operation of bulk handling systems for carbon black since an excessive amount causes plug-ups.

**Method of Test for Ash Content of Carbon Black:**

This method describes a procedure for determining the inorganic ash in carbon black.

**Method of Test for Heating Loss of Carbon Black:**

This method describes a procedure for determining the heating loss, primarily due to moisture, of carbon black.

**Method of Test for pH of Carbon Black:**

pH may be used to characterize certain types of carbon black.

**Method of Test for Iodine Adsorption Number of Carbon Black:**

The amount of iodine adsorbed by carbon blacks has frequently been used as a measure of the surface areas.

The recommendations in this report have been submitted to letter ballot of the Committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Physical Tests* (J. F. Svetlik, chairman) prepared the pellet size, sieve residue, attrition, pour density, and fines test methods.

*Subcommittee II on Chemical Tests* (L. G. Mason, chairman) submitted the test methods for ash content, heating loss, pH, and iodine number. Action on volatile content, oil absorption, solvent extractables, sulfur content, and nitrogen absorption tests was deferred.

*Subcommittee III on Optical Tests* (J. F. Ambrose, chairman) has organized task groups to investigate the discoloration of benzene by carbon black, the tinting strength of black, the jetness of blacks, and a method for the determination of the size distribution of blacks. This group is also planning to organize a carbon black sample library.

*Subcommittee V on Sampling* (R. O. Treat, chairman) is considering the problem of carbon black sampling in the light of ASTM publications on statistical sampling.

This report has been submitted to letter ballot of the committee, which consists of 41 members; 37 members returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. R. DONER,
*Chairman.*

ALFRED G. COBBE,
*Secretary.*

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE D-25

ON

# CASEIN AND SIMILAR PROTEIN MATERIALS*

Committee D-25 on Casein and Similar Protein Materials held two meetings during the year: an organization meeting in Atlantic City, N. J. on June 20, 1956, and in Philadelphia, Pa. on February 6, 1957.

The committee records with regret the death of S. B. Gilpin who so ably contributed to the organization of the committee. Eight new participants have been added to the membership of the committee.

The committee consists of 34 members, of whom 10 are classified as producers, 18 as consumers, and 6 as general interest members.

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Definitions* (M. E. Stonebreaker, chairman) is preparing a group of terms to be defined which are used in the field of industrial casein, isolated soya protein, and similar protein materials.

*Subcommittee II on Sampling* (J. C. Rice, chairman) is considering thief sampling to determine uniformity within shipments of both casein and soya protein. The study of viscosity is a first approach to the sampling problem.

*Subcommittee III on Chemical Tests* (E. G. Simpson, chairman) has under way an interlaboratory collaborative test program using the oven method of testing for moisture in casein.

*Subcommittee IV on Physical Tests* (L. E. Georgevits, chairman).—A collaborative program for the preparation of a procedure for sieve analysis is nearing completion. Work on tests for solubility and viscosity is under way.

*Subcommittee VI on Research* (L. E. Clark, Jr., chairman) is considering qualitative tests used to distinguish casein from soya protein. Quantitative methods are under consideration.

This report has been submitted to letter ballot of the committee, which consists of 34 members; 17 members returned their letter ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. W. SHADER,
*Chairman.*

L. E. GEORGEVITS,
*Secretary.*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

**Method of Test for Fines Content of Pelleted Carbon Black:**

Fines or dust are important in the operation of bulk handling systems for carbon black since an excessive amount causes plug-ups.

**Method of Test for Ash Content of Carbon Black:**

This method describes a procedure for determining the inorganic ash in carbon black.

**Method of Test for Heating Loss of Carbon Black:**

This method describes a procedure for determining the heating loss, primarily due to moisture, of carbon black.

**Method of Test for pH of Carbon Black:**

pH may be used to characterize certain types of carbon black.

**Method of Test for Iodine Adsorption Number of Carbon Black:**

The amount of iodine adsorbed by carbon blacks has frequently been used as a measure of the surface areas.

The recommendations in this report have been submitted to letter ballot of the Committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Physical Tests* (J. F. Svetlik, chairman) prepared the pellet size, sieve residue, attrition, pour density, and fines test methods.

*Subcommittee II on Chemical Tests* (L. G. Mason, chairman) submitted the test methods for ash content, heating loss, pH, and iodine number. Action on volatile content, oil absorption, solvent extractables, sulfur content, and nitrogen absorption tests was deferred.

*Subcommittee III on Optical Tests* (J. F. Ambrose, chairman) has organized task groups to investigate the discoloration of benzene by carbon black, the tinting strength of black, the jetness of blacks, and a method for the determination of the size distribution of blacks. This group is also planning to organize a carbon black sample library.

*Subcommittee V on Sampling* (R. O. Treat, chairman) is considering the problem of carbon black sampling in the light of ASTM publications on statistical sampling.

This report has been submitted to letter ballot of the committee, which consists of 41 members; 37 members returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. R. DONER,
*Chairman.*

ALFRED G. COBBE,
*Secretary.*

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

# REPORT OF COMMITTEE D-25

## ON

# CASEIN AND SIMILAR PROTEIN MATERIALS*

Committee D-25 on Casein and Similar Protein Materials held two meetings during the year: an organization meeting in Atlantic City, N. J. on June 20, 1956, and in Philadelphia, Pa. on February 6, 1957.

The committee records with regret the death of S. B. Gilpin who so ably contributed to the organization of the committee. Eight new participants have been added to the membership of the committee.

The committee consists of 34 members, of whom 10 are classified as producers, 18 as consumers, and 6 as general interest members.

### ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Definitions* (M. E. Stonebreaker, chairman) is preparing a group of terms to be defined which are used in the field of industrial casein, isolated soya protein, and similar protein materials.

*Subcommittee II on Sampling* (J. C. Rice, chairman) is considering thief sampling to determine uniformity within shipments of both casein and soya protein. The study of viscosity is a first approach to the sampling problem.

*Subcommittee III on Chemical Tests* (E. G. Simpson, chairman) has under way an interlaboratory collaborative test program using the oven method of testing for moisture in casein.

*Subcommittee IV on Physical Tests* (L. E. Georgevits, chairman).—A collaborative program for the preparation of a procedure for sieve analysis is nearing completion. Work on tests for solubility and viscosity is under way.

*Subcommittee VI on Research* (L. E. Clark, Jr., chairman) is considering qualitative tests used to distinguish casein from soya protein. Quantitative methods are under consideration.

This report has been submitted to letter ballot of the committee, which consists of 34 members; 17 members returned their letter ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. W. SHADER,
*Chairman.*

L. E. GEORGEVITS,
*Secretary.*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

# REPORT OF ALCA-ASTM JOINT COMMITTEE

# ON

# LEATHER*

The Joint ALCA-ASTM Committee on Leather was established in 1956 as authorized by the governing groups of the American Leather Chemists' Assn. and ASTM. The organization meeting was held at ASTM Headquarters in Philadelphia, Pa. on March 13, 1956. Three meetings have been held subsequently: on May 22, 1956 in Philadelphia, Pa., on Oct. 22, 1956 and Feb. 14, 1957 in New York City. The following officers were elected at the organization meeting:

Chairman, Joseph R. Kanagy, National Bureau of Standards.

Vice-Chairman, Ralph J. Wirshing, General Motors Corp.

Secretary, Malcolm H. Battles, A. C. Lawrence Leather Co.

## NEW TENTATIVES

The committee recommends for publication as tentative the following methods and definitions as appended hereto:[1]

*Tentative Method of:*

Test for Area of Leather, and
Test for Width of Leather.

*Tentative Definitions of:*

Terms Relating to Leather.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Nomenclature, Definitions, and Applicability* (R. M. Lollar, chairman) prepared the proposed Tentative Definitions of Terms Relating to the Testing of Leather, referred to earlier in this report, and reviewed test methods in regard to definitions and applicability.

*Subcommittee II on Sampling and Conditions for Physical Testing* (C. W. Mann, chairman) is preparing a proposed Tentative Method of Conditioning Leather and Leather Products for Testing.

*Subcommittee III on Physical Testing* (G. A. Butz, chairman).

*Section 2 on Physical Dimensions* (J. Naghski, chairman) prepared the methods of test for width and area of leather, referred to earlier in the report.

*Section 3 on Tensile Properties* (R. L. Young, chairman) is working on methods for tensile strength, stitch tear, double hole, breaking strength, and elongation.

*Section 4 on Resistance to Water* (R. G. Ashcraft, chairman) will study water resistance and water repellency of shoe upper leather, sole leather, glove leather, garment leather, upholstery leather, and mechanical leather. In evaluating the methods, the subcommittee will establish a statistical sampling plan, obtain samples, test them, and correlate results with actual wear.

*Section 5 on Surface Characteristics* (Robert Stubbings, chairman) is concerned with surface characteristics and surface appearances of either finished or unfinished leather. Plans have been made to study methods for cold failure, staining, bleeding and crocking, water spotting, fading, scuff, abrasion, flex, and adhesion.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 7.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

*Section 7 on Mechanical Leathers (Miscellaneous Properties)* (A. N. Compton, chairman) is preparing methods of test for corrosion produced by leather in contact with metals, and for oil or air permeability of leather cup packings.

A method to determine the porosity of leather packings, which has been approved by the National Industrial Leather Assn., is being reviewed.

*Section 8 on Deterioration Effects* (S. S. Kremen, chairman) is concerned initially with a method for determining perspiration resistance of leather. The first problem will be to decide upon a proper solution of reagents to simulate perspiration and to evaluate this solution.

*Section 9 on Bond Strength of Belting Leathers* (P. Rhodes, chairman) has recommended that Federal Specification KK-B-201c be used as a basis for a method to determine bond strength of belting leathers.

*Section 10 on Miscellaneous Physical Properties* (A. N. Kay, chairman) is considering a method of test for stiffness in leather. Stiffness is much affected by thickness and least variability is obtained when the moment is divided by $t^3$ ($t$ = thickness). However, leather when bent grain in or grain out does not follow Hooke's law. Testing machines satisfactory for relatively stiff leathers such as sole, belting, and side are not suitable for softer leathers such as garment and glove and different machines are being considered for this purpose.

*Subcommittee IV on Research* (M. M. Baldwin, chairman) plans to compile a list of current research on test methods both within the committee and by the leather industry in general. The subcommittee will also make recommendations to the Advisory Committee as to areas for research on new test methods and will arrange for the presentation of papers on research on testing methods in technical programs at meetings of the committee.

*Subcommittee V on Editorial* (L. Boor, chairman) has concerned itself principally with the proper form and organization for presentation of test methods.

This report has been submitted to letter ballot of the committee, which consists of 55 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. R. KANAGY,
*Chairman.*

M. H. BATTLES,
*Secretary.*

# REPORT OF COMMITTEE E-1
## ON
# METHODS OF TESTING*

Committee E-1 on Methods of Testing held a meeting at Society Headquarters in Philadelphia, Pa., on April 23, 1957.

Meetings were held of sixteen E-1 subcommittees and task groups during ASTM Committee Week, in Philadelphia, Pa., from February 4 to 8, 1957.

Meetings of the following subcommittees and task groups were held in Cleveland, Ohio, on November 26 and 27:

Subcommittee 17 on Thermometers,
Subcommittee 18 on Hydrometers,
Subcommittee 19 on Glassware Laboratory Apparatus,
Subcommittee 21 on Metalware Laboratory Apparatus,
Task Group (Sub. 21) on Laboratory Ovens, and
Task Group (Sub. 21) on Safety Requirements for Oxygen and Peroxide Bombs.

The Symposium on Particle Size Measurement originally planned for 1957 has been deferred and arrangements are now being made to have this symposium at the 1958 Annual Meeting of the Society in Boston, Mass., during the week of June 22 to 28.

### RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

*New Tentative Specifications for:*

Weighing and Drying Apparatus for Microchemical Analysis (E 124 – 56 T)
Apparatus for Determination of Water by Distillation (E 123 – 56 T)

*Revision of Tentative Methods of:*

Impact Testing of Metallic Materials (E 23 – 56 T)
Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77 – 52 T)

*Tentative Revision of Standard Specifications for:*

ASTM Thermometers (E 1 – 56)

Specifications E 124 were accepted by the Standards Committee on September 18, 1956, and E 123 on November 7, 1956. They appear in Parts 3 and 4 of the 1956 Supplement to the Book of ASTM Standards. The revision of Methods E 23 was accepted on September 10, 1956, and appears in Part 1 of the 1956 Supplement. The revisions of Specifications E 1 and Method E 77 were accepted on November 15, 1956, and appear in Part 5 of the 1956 Supplement.

### PROPOSED SPECIFICATIONS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Proposed Specifications for Distillation Equipment, as appended hereto.[1]

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The proposed specifications appear in the Compilation of ASTM Standards on Petroleum Products and Lubricants, December, 1957.

## NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and methods, as appended hereto:

Method for Inspection and Verification of Hydrometers,[2]

Specifications for Gravity-Convection and Forced-Ventilation Ovens,[3] and

Method of Test for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use.[2]

## REVISION OF TENTATIVES

The committee recommends revisions, as indicated, of the following tentatives and their continuation as tentative:

### Tentative Method of Verification of Testing Machines (E 4 – 55 T),[4] revise as follows:

*Section 6*.—Change the last sentence to read as follows:

If it is desired to establish the lower limit of a loading range lower than 10 per cent of the capacity of the range, the lower limit shall be verified by applying five approximately equal test loads, none of which shall differ from the lowest by more than 5 per cent.

*Section 15 (a)*.—Add the symbol "±" before 1 per cent, thus changing this section to read as follows:

15 (*a*). The error for loads within the loading range of a testing machine shall not exceed ±1 per cent.

*Section 15 (b)*.—Change to read as follows:

(*b*) In establishing the lower limit of a loading range below 10 per cent of the capacity of the range where five applications of load are required, the algebraic difference between the highest and lowest percentage error of the five test loads shall not exceed 1.0.

NOTE 2.—This means that to establish the lower limit of a loading range less than 10 per cent of the capacity of that range, the errors for the series of five readings shall not only not exceed 1 per cent but also no two errors shall differ by more than 1.0 per cent. If the minimum error in this series is −1.0 per cent the maximum error cannot exceed 0.0 per cent; if the minimum error is −0.5 per cent the maximum error cannot exceed +0.5 per cent. If the minimum error is 0.0 per cent the maximum error cannot exceed +1.0 per cent; etc.

*Section 15 (c)*.—Change to read as follows:

(*c*) In no case shall the loading range be stated as including loads below the value which is 100 times the smallest change of load which can be estimated on the load-indicating apparatus of the testing machine.

NOTE 3.—This means that in a testing machine which has graduations so spaced that estimations can be made to $\frac{1}{10}$th division the loading range could not extend downward to a load less than that corresponding to 10 divisions. If the graduations on the load-indicating scale can be estimated only to two divisions the loading range could not extend downward below the load corresponding to 200 divisions. On most machines the smallest load that can be measured is somewhere between the two examples cited.

### Tentative Method of Verification and Classification of Extensometers (E 83 – 52 T):[4]

*Section 5*.—Replace the first sentence by the following:

Extensometers are to be calibrated throughout the full range used in testing and shall be classified accordingly. They need not be calibrated beyond the range over which they are to be used.

*Section 9*.—Change to read as follows:

9. Extensometers shall be classified in accordance with the following requirements as to maximum error of indicated strain:

| | Maximum Error of Indicated Strain |
|---|---|
| Class A | 0.00001 |
| Class B-1 | 0.0001 |
| Class B-2 | 0.0002 |
| Class C | 0.001 |
| Class D | 0.01 |
| Class E | 0.1 |

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[2] The new tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 5.

[3] The letter ballot in the committee on these specifications resulted in negative votes, and in view of this fact they are being published as information only rather than as a tentative. See p. 429.

[4] 1955 Book of ASTM Standards, Part 1.

NOTE.—Class A extensometers would ordinarily be used for determining precise values of the modulus of elasticity and for delicate measurements of permanent set or very slight deviations from Hooke's Law. Class B-1 extensometers would ordinarily be used for determining values of the modulus of elasticity and for measurement of permanent set or deviations from Hooke's Law and for determining values such as the yield strength of metallic materials. Class B-2 extensometers would be used for determining yield strength of metallic materials. This classification would generally apply to extensometers having gage lengths less than 2 in. which would not meet Class B-1 requirements. Definitions of the terms "modulus of elasticity" and "yield strength" are given in The Definitions of Terms Relating to Methods of Mechanical Testing (ASTM Designation: E 6).

### Tentative Specifications for ASTM Hydrometers (E 100 – 55 T):[5]

*Table I.*—Change the over-all length of hydrometers 1H to 10H from "300 to 335 mm" to "325 to 335 mm."

Change the over-all length of hydrometers 51H to 60H and Hydrometers 71H to 74H from "375 to 400 mm" to "374 to 387 mm."

*Table II.*—In the third column, change the word "gasoline" to "*iso*-octane." Also, under requirements for hydrometers for light liquids other than alcohol, change the range of the third hydrometer from "0.700 to 1.100" to "0.700 to 1.070."

## REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following two standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

### Standard Specifications for ASTM Thermometers (E 1 – 56):[6]

*Thermometer 49C.*—Add "70 C" as a third standardization temperature.

*Thermometers 6C and 6F.*—Add the following Footnote *b* with references after "ice point" in the columns opposite "standardization":

[b] This thermometer is especially sensitive to changes in emergent column temperatures because organic liquids are used as the thermometer fluid. As a means of avoiding errors due to this cause in determining changes in bulb volume with age, periodic standardization at the ice point under total immersion conditions is acceptable. Any change in this correction should be applied to all corrections for partial immersion use including the ice point correction at partial immersion.

*Thermometer 85F.*—Add the requirement that an arrow be etched on the stem opposite the 225 F graduation.

### Standard Method of Test for Diamond Pyramid Hardness of Metallic Materials (E 92 – 55):[4]

*Section 5 (b).*—Add: "When laminated material is tested, the thickness of the individual component being tested shall be used for the thickness–diagonal length relationship."

*Section 6 (c).*—Add the following sentence at the end of this section: "When laminated material is tested, a bond surface shall be considered as an edge for spacing of indentation calculations." This revision is proposed as a result of suggestions received from Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts.

## REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the following two standards be reverted to tentative and revised as appended hereto:[7]

Method of Bend Testing for Ductility of Metals (**E 16 – 39**),[4] and
Methods of Test for Thickness of Solid Electrical Insulation (**D 374 – 42**).[8]

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[5] 1955 Book of ASTM Standards, Part 5.

[6] 1956 Supplement to Book of ASTM Standards, Part 5.

[7] The revised tentatives appear in the 1957 Supplement to Book of ASTM Standards, Parts 1 and 6 respectively.

[8] 1955 Book of ASTM Standards, Part 6.

### ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Verification of Calibration Devices for Verifying Testing Machines (**E 74 - 55 T**),[4] and

Tentative Recommended Practice for Operation of Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Artificial Weathering Test (**E 42 - 55 T**).[8]

### ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Saybolt Furol Viscosity of Asphaltic Materials at High Temperatures (E 102-54 T) be approved for reference to letter ballot of the Society for adoption of standard with revision as follows:

*Section 2.*—In Note 1, change the words "Notes 1 and 2 in" to "the Appendix of" so that the Note will read in part as follows: "Reference should be made to the Appendix of the Standard Method"....

### TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following three tentatives as consideration is now being given to changes to be recommended in these tentatives during the coming year:

Tentative Method for Determination of Young's Modulus at Room Temperature (**E 111 - 55 T**),[4]

Tentative Definitions with Procedures Relating to Conditioning and Weathering (**E 41 - 48 T**),[9] and

Tentative Methods of Test for Measuring Water Vapor Transmission of Materials in Sheet Form (**E 96 - 53 T**).[8]

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[10]

### ACTIVITIES OF SUBCOMMITTEES

An extensive summary of the activities of the subcommittees and task groups of Committee E-1 will appear in the ASTM BULLETIN subsequent to the Annual Meeting of the Society.

This report has been submitted to letter ballot of the committee, which consists of 49 voting members; 28 members returned their ballots, of whom 27 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
*Chairman.*

P. J. SMITH,
*Secretary.*

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[9] See Editorial Note, p. 428.

[10] The letter ballot vote on these recommendations was favorable (see footnote 3); the results of the vote are on record at ASTM Headquarters.

## EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Revision of Tentative Methods of:*

Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 55 T)

Tension Testing of Metallic Materials (E 8 – 54 T)

*Revision of Tentative Specifications for:*

Weighing and Drying Apparatus for Microchemical Analysis (E 124 – 56 T)

*Revision of Tentative Definitions:*

With Procedures Relating to Conditioning and Weathering (E 41 – 48 T)

The revision of Methods E 18 was accepted by the Standards Committee on November 1, 1957 and appears in the 1957 Supplement to Book of ASTM Standards, Parts 1 and 2; the revision of Methods E 8 was accepted on December 26, 1957 and appears in Part 1 of the 1957 Supplement; on December 23, 1957 the revision of Definitions E 41 was accepted which is set forth in the yellow stickers for Parts 3, 4, 5, 6 and 7 of the 1957 Supplement, as well as the revision of Specifications E 124 which appears in Parts 3, 4 and 5 of the Supplement.

# PROPOSED SPECIFICATIONS FOR

# GRAVITY-CONVECTION AND FORCED-VENTILATION LABORATORY OVENS[1,2]

These are proposed specifications and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (*a*) These specifications cover the performance requirements for general purpose air ovens ordinarily used in testing operations. They are applicable to ovens which are designed to operate over all or part of the temperature range from 20 C above ambient temperature to 300 C, and which have a testing chamber of not more than 25 cu ft (0.6 cu m).

(*b*) These specifications do not include safety requirements that are essential for ovens used in the presence of combustible vapors or gases.

### Types and Grades

2. (*a*) These specifications cover air ovens of the following two types, each type having two grades, A and B:

(*1*) *Type I*.—An oven ventilated by gravity-convection.

(*2*) *Type II*.—An oven having forced ventilation.

(*b*) Grade A ovens are required to meet higher standards of performance than Grade B ovens.

### Performance Requirements

3. (*a*) The temperature within the testing chamber shall be controllable by an automatic device, and shall be uniform within the tolerances given in Table I for the particular type and grade of oven when tested in accordance with Section 4.

(*b*) The "time constant" is an arbitrary measure of the rate at which a standard specimen is heated following the procedure prescribed in Section 5. The value of the time constant shall not exceed the maximum value given in Table I for the particular type and grade of oven.

(*c*) The rate of ventilation of the testing chamber shall be at least as great as the rate specified in Table I for the particular type and grade of oven when measured in accordance with the procedure given in Section 6.

## METHODS OF TESTING

### Temperature Uniformity

4. (*a*) Place nine calibrated thermocouples (Note 1) made from No. 24-gage iron or copper-constantan wire, having a junction size of not more than 0.1 in. (2.5 mm), in the empty testing chamber with shelves in place and vents open. Locate one thermocouple in each of the eight corners of the oven approximately 2 in. from each wall and place the ninth thermocouple within 1 in. of the

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[1] These proposed specifications are under the jurisdiction of the ASTM Committee E-1 on Methods of Testing.

[2] Published as information December, 1957.

geometric center of the chamber. A minimum length of 12 in. of lead wire for each thermocouple shall be inside the oven to minimize the conduction of heat from the thermocouple.

(*b*) Bring the oven to the desired temperature and allow it to reach a steady state (Note 2). Record the temperatures of the nine thermocouples for a period of at least 24 hr, and determine from the record the maximum deviation of each point from the desired temperature. The ambient room temperature shall not vary by more than a total of 10 C, and the line voltage for electrically heated ovens shall not vary by more than a total of 5 per cent during the test.

TABLE I.—PERFORMANCE REQUIREMENTS FOR OVENS.

| | Type I | | Type II | |
|---|---|---|---|---|
| | Grade A | Grade B | Grade A | Grade B |
| Deviation from temperature of test throughout testing chamber during 24-hr period for the following differentials between ambient and test temperatures: | | | | |
| 50 C or less, max, deg C | 1 | 2.5 | 0.5 | 1.25 |
| More than 50 C, max, per cent of differential | 2 | 5 | 1 | 2.5 |
| Time constant, max, sec | 600 | 720 | 480 | 660 |
| Rate of ventilation of testing chamber, min, air changes per hour | 10 | 10 | 100 | 100 |

NOTE 1.—If calibrated thermocouples are not available, nine thermocouples made from the same spool of wire may be used provided they give the same value for temperature when placed adjacent to one another in the testing chamber at the temperature of test.

NOTE 2.—Some ovens may require as much as 24 hr to reach a steady state. If a steady state does not exist, there is a drift in the temperature toward the steady state condition.

## Time Constant

5. (*a*) Heat the oven to the highest temperature at which the oven is to be operated. For this test, use a standard specimen consisting of a brass cylinder $\frac{3}{8}$ in. in diameter and $2\frac{1}{4}$ in. long to which one junction of a differential thermocouple has been soldered. Suspend the specimen vertically in the testing chamber by means of an asbestos cord or fine wire (No. 36 gage, max). Place the free junction of the differential thermocouple in the air space of the chamber at least 3 in. removed from the specimen.

(*b*) Open the door of the oven for 1 min while the standard specimen and differential thermocouple are being suspended in the testing chamber. Then close the door and either record or measure the temperature differential every 10 sec. The time in seconds required for the temperature difference to decrease to one-tenth of the original value (for example, from 120 C to 12 C) shall be determined and considered to be the time constant of the oven.

## Rate of Ventilation[3]

6. (*a*) Seal the ventilation ports, door, and all apertures of electrically heated ovens with adhesive tape or by other means to prevent any air from passing through the oven (Note 3). Connect a watt-hour meter, with the smallest division reading in 0.01 w-hr, in the electrical supply line to the oven.

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[3] This method is only applicable to electrically heated ovens. Methods are being developed by the committee for determining the rate of ventilation of ovens that are not electrically heated and for determining the uniformity of air flow within the testing chamber.

(*b*) Heat the oven to a temperature of 80 ± 2 C above the ambient room temperature, and while at this temperature measure the consumption of electrical energy for a period of at least $\frac{1}{2}$ hr. Start and stop the test at corresponding points of the "on-off" heating cycle, that is, at the moment when the heaters are switched on by the thermostat.

(*c*) Then remove the seals, open the ventilation ports, and measure the consumption of electrical energy in the same manner. The ambient room temperature measured at a point 6 ft from the oven, approximately level with its base and at least 2 ft from any solid object, shall be the same within 0.2 C during the two tests.

(*d*) Calculate the number of changes per hour of the air in the test chamber from the following equation:

$$N = \frac{3590\,(X - Y)}{VD\Delta T}$$

where:

$N$ = number of air changes per hour,

$X$ = average power consumption in watts during ventilation obtained by dividing the energy consumption determined from the watt-hour meter readings by the duration of the test in hours,

$Y$ = average power consumption in watts with no ventilation, computed in the same manner,

$V$ = volume of the testing chamber in cubic centimeters,

$D$ = density in grams per cubic centimeter of the ambient room air during the test, and

$\Delta T$ = difference in temperature between the testing chamber and the ambient room air in degrees Centigrade.

NOTE 3.—In forced-ventilation ovens, the space around the motor shaft where it enters the oven must be closed, but the fan speed must not be affected by the closure.

# REPORT OF COMMITTEE E-2
## ON
# EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held two meeetings during the year: in Atlantic City, N. J., on June 21, 1956, and in Pittsburgh, Pa., on March 7, 1957. The Executive Subcommittee met at these times, as well as in Detroit, Mich., on November 12, 1956.

The committee consists of 179 members. This represents an increase of nine members since the last Annual Meeting.

The committee prepared Recommendations on the Form of ASTM Spectrochemical Methods which will be available in printed form.

### SUGGESTED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends that the following eight suggested methods be published as information as appended hereto:[1]

*Suggested Methods for Spectrochemical Analysis of:*

Nickel-Base High-Temperature Alloys by the Point-to-Plane Spark Technique,
Bismuth-Cadmium Eutectic Alloy by the Powder - D-C Arc Technique,
Tin-Cadmium Eutectic Alloy by the Powder - D-C Arc Technique,
Aluminum and Its Alloys by the Point-to-Plane Spark and Intermittent Arc Techniques Using a Recording Photoelectric Spectrometer,
Magnesium and Its Alloys by the Pin-to-Pin Spark Technique,
Basic Open-Hearth Slags for Lime-Silica Ratios by the Powder-Spark Technique,
Alumina Ceramic Materials by the Powder - D-C Arc Technique, and
Cobaltic Oxide for Impurities by the Powder - D-C Arc Technique.

### NEW TENTATIVES

The committee recommends for publication as tentative the following method and recommended practice:

*Tentative Method for:*

Spectrochemical Analysis of Nickel Alloys by the Powder - D-C Arc Technique.[2]

This method has grown out of Suggested Method E-2 SM 5-1 of similar title, which appears in "Methods for Emission Spectrochemical Analysis," published in 1953 by ASTM. The method was tested cooperatively by nine laboratories acting as a task group within Subcommittee V and in cooperation with Committee F-1 on Electron Tubes and Semiconductor Devices. The supporting data are appended hereto.[3]

*Tentative Recommended Practice for:*

Designation of Sizes and Shapes of Preformed Graphite Electrodes.[2]

This practice has grown out of a desire of the committee to standardize a limited number of electrode types in order to reduce the cost and to simplify spectrochemical analytical methods. The report of the task group within Subcommittee IV which prepared this tentative practice is appended hereto.[4]

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The suggested methods appear in "Methods for Emission Spectrochemical Analysis," October 1957.

[2] Available as a separate reprint; appears also in "Methods for Emission Spectrochemical Analysis," October 1957.

[3] See p. 436.

[4] See p. 439.

## TENTATIVES CONTINUED WITHOUT REVISION

The following tentative methods, which have stood for several years without revision, were reviewed by the appropriate subcommittees, and the committee recommends that they be continued as tentative without revision:

*Tentative Method for Spectrochemical Analysis of:*

Zinc-Base Alloys and High-Grade Zinc by the Solution - D-C Arc Technique (**E 27 – 53 T**),
Tin Alloys for Minor Constituents and Impurities (**E 51 – 43 T**), and
Aluminum and Aluminum-Base Alloys by the Point-to-Plane Spark Technique (**E 101 – 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

## SYMPOSIUM

A Symposium on Spectrographic Analysis for Trace Elements consisting of the papers listed below,[6] will be sponsored by the committee at the 1957 Annual Meeting:

"Emission Spectrometric Determination of Oxygen in Metals" by V. A. Fassel, W. A. Gordon, and R. W. Tabeling, Institute for Atomic Research and Department of Chemistry, Iowa State College.
"The Determination of Trace Elements in Metals" by J. A. Norris, Oak Ridge National Laboratory.
"Trace Analysis by Means of the Graphite Spark" by James M. Morris and Francis X. Pink, Metal Hydrides, Inc.
"Medical and Biological Materials" by Bert Vallee, Harvard Medical School and Peter Bent Brigham Hospital.
"Plants and Soils" by W. G. Schrenk, Kansas State College.
"Geological Materials" by K. J. Murata, U. S. Geological Survey.

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Apparatus and Equipment* (A. J. Mittledorf, chairman) has approved a report on cooperative tests of four photographic emulsions used in spectrochemical analysis, as appended hereto.[7] A summary of the characteristics of the various emulsions used throughout the world is being compiled. Other tasks in progress include preparation of a report comparing the specifications of the various modern spectrographs available in United States and Canada and revising the suggested practice for describing and specifying electrical sources used in spectrochemical analysis.

*Subcommittee II on Fundamental Practices* (R. W. Smith, chairman) is preparing a suggested practice for the computation of the precision and accuracy of spectrographic data and has prepared a suggested practice for basic calculations in spectrochemical analysis. The subcommittee is developing a suggested recommended basic practice in flame photometry and is assembling a bibliography on flame photometry suitable for publication by ASTM. A program is in progress on testing powder specimens to determine what information should be included in a standard method of analysis by X-ray fluorescence techniques. The subcommittee is also preparing a bibliography on X-ray fluorescence and is scanning current literature with the objective of persuading authors of outstanding methods involving X-ray fluorescence to write suggested methods.

*Subcommittee III, Editorial* (K. O. Stevenson, chairman) is preparing final copy for the Recommendations on the Form of ASTM Spectrochemical Methods, and it is reviewing proposed recommendations in the field of emission spectroscopy by other ASTM committees. In preparation is a proposed tentative nomenclature which is similar to the tentative nomenclature prepared by Committee E-13 on Absorption Spectroscopy.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[6] Issued as a separate publication *ASTM STP No. 221*.

[7] See p. 442.

The subcommittee is reviewing material to be included in a revised edition of the "Methods for Emission Spectrochemical Analysis." Forty-nine practices or methods are to be retained from the 1953 edition, and thirty-two new or revised practices or methods are to be added. Of the thirty-two new manuscripts, twelve are under the jurisdiction of ASTM committees other than Committee E-2.

*Subcommittee IV on Electrodes, Pure Materials, Reagents, and Standards* (W. J. Edgar, chairman) is maintaining a current list of all standards and pure materials available throughout the world and has prepared a proposed tentative method for designation of sizes and shapes of preformed graphite electrodes.[2] It has also prepared a report[4] on the survey of Committee E-2 members regarding sizes and shapes of graphite electrodes used. A questionnaire will be circulated in the United States and Canada to determine the need for standards.

*Subcommittee V on Copper, Nickel, and Their Alloys* (A. W. Young, chairman) is assisting the National Bureau of Standards in preparing copper-alloy standard samples and is working on an effective general method for analysis of copper alloys. The subcommittee is soliciting methods pertaining to the analysis of nickel-base alloys; it has prepared a proposed tentative method for analysis of nickel-base alloys by the powder - d-c arc technique[2] and has worked with Committee F-1 to evaluate this method. A copy of the report on the cooperative test is appended hereto.[3] This group prepared the Suggested Method for Nickel-Base High-Temperature Alloys referred to earlier in the report. Suggested Method E-2 SM 5–2 (Spectrochemical Analysis of Wrought Copper Alloys by the D-C Arc Technique) is being reviewed toward establishing it as a tentative.

*Subcommittee VI on Lead, Tin, Zinc, and Related Materials* (G. L. Crumrine, chairman) prepared two suggested methods referred to earlier in the report, on Bismuth-Cadmium and on Tin-Cadmium Eutectic Alloys. A revised method combining SM 8-2, 8-3, and 8-5 will be cooperatively tested looking toward publication as tentative.

*Subcommittee VII on Aluminum and Magnesium and Their Alloys* (T. M. Hess, chairman) prepared the Suggested Methods for Aluminum and Magnesium referred to earlier in the report. The suggested method for spectrochemical analysis of magnesium-base alloys by the point-to-plane spark technique is being cooperatively tested with the intention of raising it to tentative status. A method for spectrochemical analysis of aluminum and its alloys by the point-to-plane spark technique using a clock-indicating photoelectric spectrometer is being prepared.

*Subcommittee VIII on Titanium, Zirconium, and Related Metals* (P. A. Leichtle, chairman) has prepared three suggested methods for the analysis of titanium and its alloys. A task group concerned with zirconium, hafnium, and their alloys has formulated a program for the procurement of zirconium standards. Three spectrographic methods for the determination of impurities in zirconium and hafnium are being considered for publication as suggested methods.

*Subcommittee IX on Ferrous Alloys* (G. E. Pellissier, chairman) has been working on several methods to be proposed later as tentatives. These include methods for the analysis of plain carbon and low-alloy steel by point-to-plane and rod-to-rod techniques, stainless-type steels by the point-to-plane spark technique using an ARL Quantometer, and for the spectrochemical analysis of high-alloy and stainless-type steels by the point-to-plane technique using a Baird-Dow direct-reading spectrometer. Based on replies to a questionnaire concerning methods needed for cast iron analysis, a method using a point-to-plane spark technique is being drafted. A suggested method for the determination of acid-soluble aluminum in low-alloy steel by the d-c arc solution technique has been submitted. Plans are being formulated to cooperatively test SM 9-4 (Suggested Method for Spectrochemical Analysis of Steel for Boron by the Point-to-Plane Intermittent Arc Technique) with the intention of recommending it for publication as tentative.

*Subcommittee X on Glass, Ceramics, Alkalies, and Cementitious Materials* (E. S. Hodge, chairman) prepared the Suggested Method for Alumina Ceramic Materials referred to earlier in the report. Three suggested methods concerned with the analysis of caustic soda, sodium salts, and magnesium chloride are being considered.

*Subcommittee XI on Slags, Ores, and Miscellaneous Non-Metallic Materials* (R. G. Russell, chairman) prepared the Suggested Methods for analysis of basic open-hearth slag and cobaltic oxide referred to earlier in the report. Under development are suggested methods for the analysis of blast-furnace and steel-making slags, and of iron ore. A general method for semiquantitative analysis is ready for submittal to Committee E-2. A progress report on a study of universal methods of spectrochemical analysis was published in the ASTM BULLETIN for September, 1956. The subcommittee is developing a method for the analysis of cerium minerals and is considering a method for the analysis of titanium oxide. The subcommittee reviewed seven suggested methods of analysis of diesel lubricating oil, a suggested sampling procedure, and a proposed recommended practice for applying precision data for petroleum products and lubricants. These were prepared by Committee D-2 and appear in the ASTM publication entitled "Suggested Methods for Spectrochemical Analysis of Used Diesel Lubricating Oils."

This report has been submitted to letter ballot of the committee, which consists of 179 members; 60 members returned their ballots, of whom 59 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

DAVID L. FRY
*Chairman.*

CYRUS FELDMAN,
*Secretary.*

## APPENDIX I

# REPORT OF COOPERATIVE TESTS ON PROPOSED TENTATIVE METHOD FOR SPECTROCHEMICAL ANALYSIS OF NICKEL ALLOYS BY THE POWDER - D-C ARC TECHNIQUE

Many laboratories have found the method designated as E-2 SM 5-1[1] of considerable value. It was therefore decided that it should be tested cooperatively. This report is a description of the test method and a summary of the results obtained. The investigatioe was conducted jointly by Subcommittee I-C of Committee F-1 on Materials for Specific Applications, and Subcommittee V of Committee E-2.

To test the accuracy of the method, two NBS nickel oxide standards, Nos. 671 and 672, were analyzed chemically by nine chemical and five spectrochemical laboratories. To test the reproducibility of the method a single sample, identified as Melt 95-Thermionic Nickel Cathodes, was analyzed by eight spectrochemical laboratories.

The tests show that the method has sufficient accuracy and reproducibility to be published as tentative.

In Tables I and II are given the detailed data from five spectrochemical laboratories and the averaged data from nine chemical laboratories, on the analysis of two National Bureau of Standards Nickel Oxide Standards, Nos. 671 and 672.

In Table III are given the detailed data from eight spectrochemical laboratories on the analysis of Melt 95-Thermionic Nickel Cathodes.

The chemical and spectrochemical laboratories participating in the analysis of the National Bureau of Standards Standard Samples Nos. 671 and 672 were as follows:

*Chemical Laboratories:*

National Bureau of Standards, Washington, D. C.
Western Electric Co., Hawthorne Works, Chicago, Ill.
Sylvania Electric Products, Inc., Towanda Works, Towanda, Pa.
Sylvania Electric Products, Inc., Kew Gardens, N. Y.
U. S. Metals Refining Co., Carteret, N. J.
Bell Telephone Laboratories, Inc., Murray Hill, N. J.
Wilbur B. Driver Co., Newark, N. J.
Raytheon Manufacturing Co., Newton, Mass.
Driver-Harris Co., Harrison, N. J.

*Spectrochemical Laboratories:*

National Bureau of Standards, Washington, D. C.
W. B. Coleman and Co., Philadelphia, Pa.
Bell Telephone Laboratories, Inc., Murray Hill, N. J.
Raytheon Manufacturing Co., Newton, Mass.
Westinghouse Electric Co., Elmira, N. Y.

The spectrochemical laboratories participating in the analysis of Melt 95-Thermionic Nickel Cathodes were as follows:

Westinghouse Electric Co., Elmira, N. Y.
National Research Corp., Cambridge, Mass.
W. B. Coleman and Co., Philadelphia, Pa.
CBS-Hytron, Newburyport, Mass.
Dumont Laboratories, East Paterson, N. J.
Raytheon Manufacturing Co., Newton, Mass.
W. B. Driver Co., Newark, N. J.
Bell Telephone Laboratories, Murray Hill, N. J.

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[1] "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats., p. 48 (1953).

TABLE I.—ANALYSES OF NATIONAL BUREAU OF STANDARDS NICKEL OXIDE STANDARD NO. 671.
NOTE.—Average percentage deviation for all elements equals ±3.6.

| Laboratory | Percentage of Element in Nickel Oxide | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Co | Cu | Fe | Mg | Mn | Si | Ti | Al | Cr |
| Spectrochemical: | | | | | | | | | |
| A | 0.32 | 0.20 | 0.38 | 0.031 | 0.130 | ... | 0.025 | 0.0092 | ... |
| B | 0.32 | 0.15 | 0.39 | 0.032 | 0.115 | ... | 0.026 | 0.011 | 0.024 |
| C | 0.32 | 0.198 | 0.42 | 0.031 | 0.134 | 0.052 | 0.023 | 0.0092 | 0.030 |
| D | 0.32 | 0.165 | 0.40 | 0.026 | 0.123 | 0.044 | 0.024 | 0.0084 | 0.025 |
| E | 0.26 | ... | 0.39 | ... | 0.13 | ... | ... | ... | ... |
| Average | 0.308 | 0.178 | 0.396 | 0.030 | 0.126 | 0.048 | 0.0245 | 0.0094 | 0.0263 |
| Average of nine chemical laboratories | 0.304 | 0.194 | 0.390 | 0.0293 | 0.128 | 0.0472 | 0.0240 | 0.0091[a] | 0.0238 |
| Deviation from chemical laboratories | +0.004 | −0.016 | +0.006 | +0.0007 | −0.002 | +0.0008 | +0.0005 | +0.0003 | +0.0025 |
| Percentage deviation | +1.3 | −8.3 | +1.5 | +2.4 | −1.6 | +1.7 | +2.1 | +3.3 | +10.5 |
| National Bureau of Standards recommended value | 0.31 | 0.20 | 0.39 | 0.030 | 0.13 | 0.047 | 0.024 | 0.009 | 0.024 |

[a] Only one chemical laboratory reported on aluminum.

TABLE II.—ANALYSES OF NATIONAL BUREAU OF STANDARDS NICKEL OXIDE STANDARD NO. 672.
NOTE.—Average percentage deviation for all elements equals ± 5.4.

| Laboratory | Percentage of Element in Nickel Oxide | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Co | Cu | Fe | Mg | Mn | Si | Ti | Al | Cr |
| Spectrochemical: | | | | | | | | | |
| A | 0.55 | 0.017 | 0.077 | 0.019 | 0.093 | 0.096 | 0.0097 | 0.0035 | ... |
| B | 0.53 | 0.017 | 0.085 | 0.023 | 0.084 | ... | 0.0078 | 0.005 | < 0.005 |
| C | 0.56 | 0.018 | 0.075 | 0.020 | 0.093 | 0.10 | 0.0082 | 0.0047 | < 0.005 |
| D | 0.63 | 0.018 | 0.078 | 0.018 | 0.084 | 0.098 | 0.0106 | 0.0044 | 0.0032 |
| E | 0.48 | 0.018 | 0.079 | ... | 0.086 | ... | ... | ... | ... |
| Average | 0.550 | 0.0176 | 0.0790 | 0.0200 | 0.088 | 0.098 | 0.0091 | 0.0044 | 0.0032 |
| Average of nine chemical laboratories | 0.548 | 0.0178 | 0.0790 | 0.0197 | 0.0962 | 0.112 | 0.0092 | 0.0037[a] | 0.0029 |
| Deviation from chemical laboratories | +0.002 | −0.0002 | 0.0000 | +0.0003 | −0.0074 | −0.014 | −0.0001 | +0.0007 | +0.0003 |
| Percentage deviation | +0.40 | −1.1 | 0.0 | +1.5 | −7.7 | −14.3 | −1.1 | +19.0 | +3.3 |
| National Bureau of Standards recommended value | 0.55 | 0.018 | 0.079 | 0.020 | 0.096 | 0.11 | 0.009 | 0.004 | 0.003 |

[a] Only one chemical laboratory reported on aluminum.

TABLE III.—ANALYSES OF MELT 95-THERMIONIC NICKEL CATHODES.

NOTE.—Average percentage deviation from the mean for all elements equals ±6.2.

| | Silicon | | Cobalt | | Manganese | |
|---|---|---|---|---|---|---|
| | Per Cent | Per Cent Deviation | Per Cent | Per Cent Deviation | Per Cent | Per Cent Deviation |
| Spectrochemical laboratories: | | | | | | |
| 1 | 0.023 | −14.8 | 0.100 | −3.0 | 0.027 | −12.9 |
| 2 | 0.024 | −11.1 | 0.104 | 1.0 | 0.029 | −6.4 |
| 3 | 0.027 | 0.0 | 0.097 | −5.8 | 0.028 | −9.7 |
| 4 | 0.028 | 3.7 | 0.103 | 0.0 | 0.033 | 6.4 |
| 5 | 0.033 | 18.5 | 0.100 | −3.0 | ... | ... |
| 6 | 0.025 | −7.4 | 0.105 | 1.9 | 0.030 | −3.2 |
| 7 | 0.027 | 0.0 | 0.095 | −6.8 | 0.034 | 9.7 |
| 8 | 0.029 | 7.4 | 0.091 | −11.6 | 0.031 | 0.0 |
| Average | 0.027 | ±7.8 | 0.103 | ±3.9 | 0.031 | ±6.9 |

| | Aluminum | | Iron | | Titanium | |
|---|---|---|---|---|---|---|
| | Per Cent | Per Cent Deviation | Per Cent | Per Cent Deviation | Per Cent | Per Cent Deviation |
| Spectrochemical laboratories: | | | | | | |
| 1 | ... | ... | 0.041 | −6.8 | 0.026 | −7.1 |
| 2 | 0.0087 | 6.1 | 0.041 | −6.8 | 0.028 | 0.0 |
| 3 | 0.0078 | −4.9 | 0.047 | 6.8 | 0.027 | −3.6 |
| 4 | 0.0084 | 2.4 | 0.046 | 4.5 | 0.030 | 7.1 |
| 5 | 0.008 | −2.4 | 0.042 | −4.5 | 0.025 | −10.7 |
| 6 | ... | ... | 0.043 | −2.3 | 0.026 | −7.1 |
| 7 | 0.0087 | 6.1 | 0.048 | 9.4 | 0.028 | 0.0 |
| 8 | 0.008 | −2.4 | 0.045 | 2.3 | 0.030 | 7.1 |
| Average | 0.0082 | ±4.9 | 0.044 | ±5.4 | 0.028 | ±5.3 |

| | Copper | | Manganese | | Chromium | |
|---|---|---|---|---|---|---|
| | Per Cent | Per Cent Deviation | Per Cent | Per Cent Deviation | Per Cent | Per Cent Deviation |
| Spectrochemical laboratories: | | | | | | |
| 1 | 0.013 | 4.0 | 0.099 | −5.7 | ... | ... |
| 2 | 0.013 | 4.0 | 0.102 | −2.9 | 0.0034 | 3.0 |
| 3 | 0.012 | −4.0 | 0.110 | 4.8 | 0.003 | −9.4 |
| 4 | 0.011 | −12.0 | 0.110 | 4.8 | ... | ... |
| 5 | 0.014 | 12.0 | 0.103 | −1.9 | 0.004 | 21.0 |
| 6 | 0.011 | −12.0 | 0.104 | −1.0 | 0.003 | −9.4 |
| 7 | 0.012 | −4.0 | 0.102 | −2.9 | 0.003 | −9.4 |
| 8 | 0.014 | 12.0 | 0.110 | 4.8 | ... | ... |
| Average | 0.0125 | ±8.0 | 0.105 | ±3.6 | 0.0033 | ±10.4 |

## APPENDIX II

# A SURVEY ON SIZES AND SHAPES OF GRAPHITE ELECTRODES

A Report by Subcommittee IV on Electrodes and Pure Materials

Emission spectrographers employ a large variety of sizes and shapes of graphite electrodes to perform analyses on samples in various forms. These electrodes are used when samples are prepared in powder, solution, and particle form, or when solids are sparked as flat specimens.

A few years ago it was noted that an increasing number of different sizes were required for this analytical work. It was thought that if the sizes and shapes of electrodes were standardized, a smaller variety of these graphite electrode types could be produced which would decrease the cost per electrode. Also, it would make it possible to designate by letter and number the specific size and shape of the electrode used in ASTM procedures.

For these reasons Subcommittee IV on Standards and Pure Materials was requested to study the available sizes and shapes of graphite electrodes and recommend a group which could be used throughout the spectrochemical field.

## Conclusions

Several surveys were conducted among the personnel of spectrochemical laboratories in this country to ascertain the quantity of each size and shape of graphite electrodes employed in their work. It was found that the needs of almost all laboratories could be satisfied by 29 different electrode types. Eight electrode types filled over 80 per cent of all electrode needs. A proposed Tentative Recommended Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes has been written.[1] This practice includes all 29 sizes and shapes. Table I lists the uses of 25 of the various electrodes and the relative number of users of each type.

## Activities of Task Group

Several surveys were necessary in order to complete the assignment. The first survey made in 1952 indicated there were hundreds of different sizes and shapes of electrodes being employed. However, there were only slight differences in dimensions among many of the electrodes. By combining these specifications the number was reduced to 100.

Another survey was made in 1954 to determine the total quantity of each type used. Only the 29 types listed in the Recommended Practice were found to be used to any extent. The most common electrode length used was found to be 1.5 in. and thus all electrodes were standardized to this length. This length fits into standard electrode holders and requires a minimum amount of graphite rod for production. The electrodes were also grouped by letters into five classifications as follows: *C*, *D*, *P*, *PC*, and *S*.[1]

A final survey was made in January, 1956. This survey disclosed the number

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[1] Available as a separate reprint; appears also in "Methods for Emission Spectrochemical Analysis," October 1957.

TABLE I.—TALLY OF PREFORMED ELECTRODE USE IN VARIOUS LABORATORIES.

| Electrode Use | Electrode Designation | | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | C-1 | C-2 | C-3 | C-4 | C-5 | C-6 | C-7 | C-8 | S-2 | S-3 | S-4 | S-5 | S-6 | S-7 | S-8 | S-9 | S-10 | S-11 | S-12 | S-13 | S-14 | P-1 | P-2 | D-1 | D-2 |
| I Source Type: | | | | | | | | | | | | | | | | | | | | | | | | | |
| (a) Feussner, synchronous interrupted spark | | 3 | | | 8 | | | | | | | | 1 | | 1 | | | | | | | | | 5 | 2 |
| (b) Air-gap controlled spark | | 5 | 3 | 2 | 8 | 2 | | | 1 | | 1 | | | | 4 | | 1 | 2 | 2 | 3 | | 4 | | 5 | |
| (c) Triggered, condensed low-voltage spark | 1 | 13 | 3 | 1 | 12 | 2 | 1 | 4 | | | | | 1 | | | | | | | | | | 1 | 10 | 6 |
| (d) High-voltage a-c arc | 1 | 2 | 1 | | 6 | 1 | | | | | | | | 1 | | 1 | | | 1 | 2 | | | | | |
| (e) Low-voltage a-c arc | | | | 1 | 1 | | 1 | 1 | | | | | | | 1 | | | | | | | | | | |
| (f) Triggered low-voltage a-c arc | | 1 | 1 | | 1 | 1 | | | | | | 1 | | | 2 | | | | | 3 | | | | | |
| (g) Triggered unidirectional low-voltage arc | 2 | | 1 | | 7 | 1 | 1 | 2 | 3 | 1 | 2 | | 3 | 2 | 4 | | | 3 | 4 | 10 | 4 | 6 | 1 | 1 | 4 |
| (h) D-C arc | 7 | | 2 | | 4 | 6 | 1 | 5 | | | | | | | | | | | | | | | 3 | | |
| II Material Type: | | | | | | | | | | | | | | | | | | | | | | | | | |
| (a) Ferrous | | | | | | | | | | | | | | | | | | | | | | | | | |
| (1) Alloy irons | 1 | 8 | 1 | | 10 | | | 2 | | | | | | | 2 | | | | 2 | 2 | | | 1 | 1 | |
| (2) Irons | 2 | 7 | | 1 | 10 | 2 | | 2 | | | | | | | 2 | | | | 1 | 3 | 1 | | 1 | 2 | |
| (3) Low alloy steels | 2 | 10 | 1 | 1 | 13 | 2 | 1 | 2 | | | | | | | 2 | | | | | 2 | | | 2 | 2 | |
| (4) High alloy steels | 1 | 8 | | 1 | 10 | | | 2 | | | | | | | 2 | | | | | 2 | | | 1 | 2 | |
| (5) Tool steels | 1 | 7 | | 1 | 8 | 1 | | 1 | | | | | | | 2 | | | | | 2 | 1 | | 1 | 1 | |
| (b) Non-Ferrous | | | | | | | | | | | | | | | | | | | | | | | | | |
| (1) Aluminum base | 1 | 12 | 2 | | 19 | 1 | | 2 | | | | | | | 2 | | | | 1 | 3 | 1 | 1 | 2 | 3 | |
| (2) Copper base | 3 | 10 | 1 | 1 | 9 | 2 | | 1 | | | | | | | 4 | | | | 2 | 2 | 1 | | 1 | 2 | |
| (3) High-temperature alloy | 1 | 6 | | | 4 | | | 1 | | | | | | | 2 | | | | | 2 | | | 1 | 1 | |
| (4) Magnesium base | 1 | 7 | 1 | | 10 | | | 1 | | | | | | | 2 | | | | | 1 | | | | 1 | |
| (5) Titanium base | 1 | 2 | | | 4 | | | 1 | | | | | | | 2 | | | | | 2 | | | | 1 | |
| (6) Zinc base | 1 | 7 | | | 14 | | | 1 | | | | | | | 4 | | | | 1 | 1 | | | 1 | 1 | |
| (7) Other | 1 | 4 | | | 4 | 3 | | 1 | 2 | 1 | | | | | | | 1 | 1 | | 1 | | 1 | | | |
| (c) Non-Metallic | | | | | | | | | | | | | | | | | | | | | | | | | |
| (1) Ceramic | 3 | | 2 | 1 | 5 | 2 | | 6 | 2 | 1 | | 1 | | | 4 | | | 2 | 2 | 6 | 1 | 1 | | 1 | |
| (2) Organic | 2 | 3 | 1 | 1 | 1 | 3 | | 4 | | | | | | | 3 | 1 | | | 1 | 3 | | | 1 | 2 | 1 |
| (3) Organic ash | 2 | 1 | 3 | 1 | 7 | 2 | 1 | 8 | | | | 1 | | 2 | 5 | | 1 | 2 | 3 | 11 | 1 | | 2 | 2 | |
| (4) Petroleum products | 2 | 7 | 1 | 2 | 9 | 3 | 1 | 3 | | | | | | | 5 | | | 1 | 1 | 3 | | | 2 | 14 | 9 |
| (5) Slags | 2 | | 1 | | 4 | | 1 | 3 | | | | 1 | | 1 | 1 | | | | 3 | 5 | 1 | 2 | 1 | 1 | |
| (6) Other | 5 | | 1 | | 6 | 3 | | 2 | 1 | | 2 | | 1 | | 3 | 1 | | 1 | 1 | 2 | 1 | 2 | | 2 | |
| III Material State: | | | | | | | | | | | | | | | | | | | | | | | | | |
| (a) Flats | 1 | 13 | 1 | 1 | 21 | 2 | | | | | | | | | | | | | | | | | | | |
| (b) Pellets | 1 | 4 | | 1 | 7 | 1 | | 1 | 1 | | | | | | 3 | | | | | 3 | | | | | |
| (c) Pins | | 3 | 1 | | 6 | | | | | | | | | | | | | | | | | | | | |
| (d) Powders | 7 | 3 | 2 | 2 | 11 | 4 | 4 | 10 | 3 | 1 | 2 | | 3 | 3 | 6 | 1 | 2 | 6 | 6 | 18 | 2 | 7 | 5 | 1 | |
| (e) Solutions (liquids) | | 7 | 4 | 1 | 10 | 2 | 1 | 1 | | | | | 1 | | 3 | | | | | | | 1 | | 18 | 6 |
| (f) Solutions (dried) | 1 | 1 | 4 | | 6 | 5 | 1 | 5 | | | 1 | 1 | 2 | 1 | 4 | | | 2 | 5 | 6 | 1 | 2 | 3 | 1 | 2 |

of laboratories using each type and the conditions under which each type was used. Table I lists the results of this last survey. Two of the electrode types did not receive any votes and are not included in this table. The reason for the absence of votes was that, at this time, there was still controversy over the exact specification of these electrode types. Agreement concerning the specifications was finally reached, and the dimensions are listed under PC-1 and PC-2.[1]

## RECOMMENDATIONS

It is the recommendation of Subcommittee IV that the Recommended Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes[1] be accepted by ASTM for publication as tentative. It is further recommended that an attempt be made to establish a standard practice for the specification of other physical aspects of graphite electrodes, such as porosity, density, electrical conductivity, and purity.

## APPENDIX III

# COMPARISON OF PHOTOGRAPHIC EMULSIONS SA NO. 2, 103-0, AND 33 WITH SA NO. 1 EMULSION

### Progress Report by Subcommittee I on Equipment

This emulsion testing program, which has been in progress for several years, was motivated by a desire on the part of the spectrographers of Committee E-2 to reduce and improve the number of emulsions used today. One supplier had suggested to the Society that if a few of the better emulsions could be selected, the supplier could discontinue the manufacture of the majority of emulsions and spend more time improving these few.

To accomplish its task, Subcommittee I first devised a method for testing emulsions. Using this method it tested four common emulsion types cooperatively. When more have been tested, they expect to be able to show the interchangeability between some emulsions and to choose the better types for recommended standards.

In February, 1953, eight cooperating members of Committee E-2 undertook to test four spectrographic emulsions in order to evaluate and compare them for routine and special spectrochemical problems. Eastman Kodak emulsions SA No. 1, SA No. 2 and 103-0 were tested on plates and 35-mm film; the 33 emulsion on plates only (33 is available only in plates). The testing procedure devised with the cooperation of Eastman Kodak Co. was designed to obtain the following information:

1. Gamma *versus* wavelength relationships in the ultraviolet,
2. Speed ratios of the emulsions as compared to SA No. 1, and
3. General physical properties of the emulsions such as graininess, resolution, curling tendencies of film, etc.

Finally, each participant was asked whether, on the basis of his tests, he would rate one of the emulsions superior to the others for qualitative and quantitative spectrographic work, or both. The characteristics to be considered were as follows:

1. Long wavelength coverage,
2. High sensitivity,
3. Reasonably high gamma (contrast),
4. Low chemical fog, and
5. Good physical properties.

The following members cooperated in the testing of plates:

R. W. Callon, Aluminium Laboratories, Ltd., Arvida, Que. (Hilger Medium Quartz spectrograph)

R. E. Beach, Eastman Kodak Co., Rochester, N. Y. (Hilger Large Littrow spectrograph)

Sol Goldspiel, New York Naval Shipyard, Brooklyn, N. Y. (Jarrell-Ash 3.4-m grating spectrograph)

W. O. Mansfield, Philco Tube Development Co., Lansdale, Pa. (Bausch & Lomb Large Littrow spectrograph)

The following cooperated in the testing of film:

R. R. Foster, du Pont Experiment Station, Wilmington, Del. (Jarrell-Ash 2.5-m grating spectrograph)

E. S. Hodge, Mellon Institute, Pittsburgh, Pa. (Applied Research Laboratories 2-m grating spectrograph)

TABLE I.—GAMMA VERSUS WAVELENGTH FOR SA NO. 2 EMULSION.

| Approximate Wavelength, Å | Plates | | | | Film | | | |
|---|---|---|---|---|---|---|---|---|
| | Lab. 1 | Lab. 2 | Lab. 3 | Lab. 4 | Lab. 5 | Lab. 6 | Lab. 7 | Lab. 8 |
| 2400 | 0.50 | ... | 0.67 | 0.90 | ... | 1.38 | ... | ... |
| 2500 | 0.50 | ... | 0.82 | 0.92 | ... | ... | ... | 0.67 |
| 2600 | 0.60 | ... | ... | ... | ... | 1.32 | ... | 1.75 |
| 2700 | 0.60 | ... | 0.88 | 0.90 | 2.19 | 1.27 | ... | ... |
| 2800 | 0.60 | ... | 0.83 | 0.92 | ... | ... | 1.15 | ... |
| 2900 | 0.61 | 0.55 | 0.82 | 0.94 | 3.38 | 1.33 | 1.32 | 0.84 |
| 3000 | 0.70 | 0.60 | 0.80 | 0.95 | 3.49 | ... | 1.19 | 1.19 |
| 3100 | ... | 0.63 | ... | 0.94 | 1.75 | 1.05 | 1.11 | 1.28 |
| 3200 | ... | ... | ... | ... | ... | 1.11 | ... | ... |
| 3300 | 0.82 | 0.69 | 0.97 | 0.93 | ... | 1.25 | 1.07 | 0.97 |
| 3400 | ... | ... | ... | ... | 2.05 | ... | 0.93 | ... |
| 3500 | ... | ... | ... | ... | 2.05 | ... | 1.05 | ... |
| 3600 | ... | ... | 1.31 | 1.00 | ... | ... | ... | 1.30 |
| 3700 | 1.00 | 0.74 | ... | 0.95 | 2.90 | 1.61 | 1.17 | ... |
| 3800 | ... | 0.74 | ... | 1.04 | 2.67 | ... | 1.17 | ... |
| 3900 | ... | 1.19 | 1.45 | ... | ... | 2.31 | 1.48 | ... |
| 4000 | 1.24 | ... | 1.56 | 1.13 | 3.73 | ... | 1.37 | 1.60 |
| 4100 | ... | 0.67 | ... | ... | 4.17 | ... | 1.37 | ... |
| 4200 | ... | ... | ... | ... | ... | ... | ... | ... |
| 4300 | 1.42 | 1.23 | 1.66 | ... | ... | ... | 1.88 | 1.48 |

TABLE II.—GAMMA VERSUS WAVELENGTH FOR 103-0 EMULSION.

| Approximate Wavelength, Å | Plates | | | | Film | | |
|---|---|---|---|---|---|---|---|
| | Lab. 1 | Lab. 2 | Lab. 3 | Lab. 4 | Lab. 5 | Lab. 6 | Lab. 7 |
| 2200 | ... | ... | ... | ... | ... | 0.89 | ... |
| 2300 | ... | ... | ... | ... | ... | ... | ... |
| 2400 | 1.00 | ... | 1.00 | 1.09 | ... | 1.89 | ... |
| 2500 | 1.30 | ... | 1.26 | 1.22 | ... | ... | ... |
| 2600 | 1.30 | ... | ... | 1.16 | ... | 1.60 | ... |
| 2700 | 1.30 | ... | 1.11 | ... | 2.19 | 1.41 | ... |
| 2800 | 1.22 | ... | 1.06 | 1.13 | ... | ... | 1.01 |
| 2900 | 1.34 | 0.92 | 1.15 | 1.10 | 2.75 | 1.39 | 1.42 |
| 3000 | 1.32 | 1.02 | 1.23 | 1.13 | 3.49 | ... | 1.37 |
| 3100 | 1.36 | 1.06 | 1.27 | 1.17 | 1.77 | 2.49 | 1.21 |
| 3200 | 1.33 | ... | ... | 1.15 | ... | 1.50 | ... |
| 3300 | 1.54 | 1.32 | 1.33 | ... | ... | 2.30 | 1.17 |
| 3400 | ... | ... | ... | 1.15 | 1.92 | 2.00 | 1.32 |
| 3500 | ... | ... | ... | 1.21 | ... | ... | 1.19 |
| 3600 | 1.56 | ... | 1.44 | 1.28 | 2.30 | ... | ... |
| 3700 | ... | 0.98 | ... | ... | 3.08 | 1.73 | 1.21 |
| 3800 | ... | 0.86 | ... | 1.30 | 3.08 | ... | 1.27 |
| 3900 | ... | 1.21 | 1.59 | ... | ... | 1.82 | 1.53 |
| 4000 | 1.63 | ... | ... | 1.28 | 3.49 | ... | 1.42 |
| 4100 | ... | 1.21 | 1.80 | ... | 4.70 | ... | 1.53 |
| 4200 | ... | ... | ... | ... | 3.17 | ... | ... |
| 4300 | 1.58 | 1.31 | 1.73 | ... | ... | ... | 2.35 |

- Cyrus Feldman and J. Y. Ellenburg, Oak Ridge National Laboratory, Oak Ridge, Tenn. (Applied Research Laboratories 2-m grating spectrograph)
- J. H. Coulliette, University of Chattanooga, Chattanooga, Tenn. (Applied Research Laboratories 1.5-m grating spectrograph)

## TEST PROCEDURE

### *Exposures:*

For the determination of gamma values and speed ratios, the emulsion was exposed to a suitable, constant source such as a household sun lamp, Type RS, manufactured by General Electric Co. A series of 15 to 40 exposures was made, each exposure being longer than the preceding one by a factor of 2. The series began with an exposure of 1 sec. The slit-to-source distance and the optics were so arranged that in these exposure times densities were recorded which could be measured on a microphotometer. In order to minimize variations, each laboratory exposed all of its emulsions within one day.

The emulsions were developed under identical conditions—3 min in fresh Eastman D-19 developer at 70 F with constant agitation.

### *Calculations:*

The densities were measured on a microphotometer at approximately 100-Å intervals over the range of 2250 to 4350 Å. For each wavelength an H and D (Hurter-Driffield) curve was obtained by plotting density as ordinate against the logarithm of the exposure time as abscissa.

The gamma of each H and D curve was obtained. This gamma is defined as the tangent of the angle that the straight-line portion of the H and D curve makes with the abscissa.

TABLE III.—GAMMA VERSUS WAVELENGTH FOR TYPE 33 EMULSION PLATES.

| Approximate Wavelength, Å | Laboratory 1 | Laboratory 2 | Laboratory 3 | Laboratory 4 |
|---|---|---|---|---|
| 2300 | ... | ... | ... | 0.49 |
| 2400 | 1.00 | ... | 0.76 | 1.05 |
| 2500 | 1.1 | ... | 1.02 | 1.14 |
| 2600 | 1.1 | ... | 0.91 | 1.11 |
| 2700 | 1.1 | ... | 0.96 | 1.09 |
| 2800 | ... | ... | 0.94 | 1.15 |
| 2900 | 1.0 | 0.73 | 0.88 | 0.94 |
| 3000 | 0.96 | 0.71 | 0.97 | 0.96 |
| 3100 | 0.89 | 0.81 | 0.97 | 1.01 |
| 3200 | ... | ... | ... | ... |
| 3300 | 1.12 | 0.85 | 1.11 | 1.00 |
| 3400 | ... | ... | ... | 1.11 |
| 3500 | ... | ... | ... | 1.17 |
| 3600 | ... | ... | 1.40 | ... |
| 3700 | 1.39 | 0.89 | ... | 1.20 |
| 3800 | ... | 0.70 | ... | 1.29 |
| 3900 | ... | 1.20 | 1.58 | ... |
| 4000 | 1.48 | ... | ... | 1.26 |
| 4100 | ... | 1.23 | 1.76 | ... |
| 4200 | ... | ... | ... | ... |
| 4300 | 1.53 | 1.48 | 2.05 | ... |

Speed ratios of each emulsion as compared to SA No. 1 were calculated at 2500, 3000, 3600, 4000, and 4300 Å. Two different density levels were chosen for comparison, 0.3 and 1.0. For this test emulsions on plates were compared with SA No. 1 on plates while the emulsions on film were compared with SA No. 1 on film. Using the H and D curve obtained above the speed ratios were calculated as follows:

$$\text{Speed ratio} = \frac{t_1}{t_2},$$

where:

$t_1$ = the time required for the reference emulsion to reach a given density at a given wavelength, and

$t_2$ = the time required for the test emulsion to reach the same density at the same wavelength, the two emulsions being exposed, developed, and microphotometered under identical conditions.

To illustrate, if at 2500 Å it took the reference emulsion $x$ 16 sec to reach the

### TABLE IV.—SPEED RATIO DATA FOR PLATES.

| Emulsion | Wave-length, Å | Speed Ratio | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Laboratory 1 | | Laboratory 2 | | Laboratory 3 | | Laboratory 4 | |
| | | D = 0.3 | D = 1.0 | D = 0.3 | D = 1.0 | D = 0.3 | D = 1.0 | D = 0.3 | D = 1.0 |
| SA No. 2 / SA No. 1 | 2500 | . . . | . . . | . . . | . . . | 0.36 | 0.12 | 0.3 | 1.0 |
| | 3000 | 0.46 | 0.13 | 0.25 | 0.06 | 0.46 | 0.18 | 0.4 | 0.2 |
| | 3600 | 0.6 | 0.17 | 0.4 | . . . | 0.63 | 0.23 | 0.8 | 0.3 |
| | 4000 | 1.0 | 0.37 | 0.8 | 0.1 | 1.0 | 0.46 | 1.0 | 0.4 |
| | 4300 | 2.6 | 1.0 | 1.7 | 0.6 | 2.5 | 1.2 | . . . | . . . |
| 103-0 / SA No. 1 | 2500 | . . . | . . . | . . . | . . . | 4.2 | 2.5 | 6 | 4 |
| | 3000 | 7.2 | 4.2 | 7.4 | 4.6 | 8.7 | 5.9 | 6 | 4 |
| | 3600 | 11 | 4.7 | 23 | 9 | 11 | 5.8 | 8 | 4 |
| | 4000 | 15 | 6.5 | 16 | 7.5 | 19 | 9.5 | . . . | . . . |
| | 4300 | 32 | 15 | . . . | 16 | 43 | 22 | . . . | . . . |
| 33 / SA No. 1 | 2500 | . . . | 1.2 | . . . | . . . | 1.8 | 0.9 | 2 | 1 |
| | 3000 | 2.0 | 0.8 | 1.6 | 0.6 | 1.7 | 0.8 | 2 | 0.8 |
| | 3600 | 2.8 | 1.4 | 3.0 | 1.0 | 2.5 | 1.5 | 3 | 2 |
| | 4000 | 4.9 | 2.1 | 4.7 | 2.0 | 3.5 | 2.0 | 4 | 2 |
| | 4300 | 10 | 5.1 | 9.2 | 4.6 | 9.0 | 5.1 | . . . | . . . |

### TABLE V.—SPEED RATIO DATA FOR FILM.

| Emulsion | Wave-length, Å | Speed Ratio | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Laboratory 5 | | Laboratory 6 | | Laboratory 7 | | Laboratory 8 | |
| | | D = 0.3 | D = 1.0 | D = 0.3 | D = 1.0 | D = 0.3 | D = 1.0 | D = 0.3 | D = 1.0 |
| SA No. 2 / SA No. 1 | 2500 | . . . | . . . | 0.7 | 0.5 | . . . | . . . | 1.9 | 1.0 |
| | 3000 | 1.8 | 1.0 | 1.2 | . . . | 2.5 | 1.7 | 2.1 | 1.3 |
| | 3600 | 2.7 | 1.8 | 1.0 | 1.4 | 4.3 | 2.1 | 2.4 | 1.6 |
| | 4000 | 6.8 | 2.1 | 4.0 | 3.0 | 6.7 | 3.1 | 3.6 | 2.5 |
| | 4300 | 8.0 | . . . | . . . | . . . | 18.0 | 9.6 | . . . | . . . |
| 103-0 / SA No. 1 | 2500 | . . . | . . . | 4.1 | 4.0 | . . . | . . . | . . . | . . . |
| | 3000 | 4.0 | 2.0 | 10.0 | . . . | 6.9 | 4.5 | . . . | . . . |
| | 3600 | 5.8 | 4.3 | 7.9 | 10.9 | 11.0 | 5.7 | . . . | . . . |
| | 4000 | 10.4 | 4.0 | 4.1 | 5.0 | 16.0 | 8.0 | . . . | . . . |
| | 4300 | 18.0 | . . . | . . . | . . . | 20.0 | 34 | . . . | . . . |

### TABLE VI.—SPEED RATIOS WEIGHTED AVERAGES OF DATA SUBMITTED.

| Density | Wavelength, Å | Plates | | | Film | |
|---|---|---|---|---|---|---|
| | | SA No. 2 / SA No. 1 | 103-0 / SA No. 1 | 33 / SA No. 1 | SA No. 2 / SA No. 1 | 103-0 / SA No. 1 |
| 0.3 | 2500 | 0.3 | 5 | 1.9 | 1 | 4 |
| | 3000 | 0.5 | 7 | 1.8 | 2 | 7 |
| | 3600 | 0.7 | 10 | 2.8 | 3 | 8 |
| | 4000 | 1.0 | 17 | 4.3 | 5 | 13 |
| | 4300 | 2.5 | 37 | 9.4 | 9 | 20 |
| 1.0 | 2500 | 0.1 | 3 | 1.0 | 1.0 | 4 |
| | 3000 | 0.2 | 5 | 0.8 | 1.0 | 3 |
| | 3600 | 0.25 | 6 | 1.5 | 1.5 | 6 |
| | 4000 | 0.4 | 8 | 2.0 | 2.7 | 8 |
| | 4300 | 1.1 | 18 | 5.1 | 10 | 30 |

density of 0.3 and if it took emulsion *y* only 4 sec to reach the same density at the same wavelength, then the speed ratio of emulsion *y* to emulsion *x* is $\frac{16}{4}$ or 4. This means that emulsion *y* is four times as fast as the reference emulsion *x*.

## TEST RESULTS

### *Gamma versus Wavelength:*

The data showing the gamma variation with wavelength are given in Tables I, II, and III.

Although detailed instructions were given to the cooperators in order to insure consistency of results, gamma values were found to vary considerably from laboratory to laboratory. This is not surprising in view of recent studies by Kniseley and Fassel,[1] Brech and McNally,[2] and Feldman.[3]

In all of these studies it is reported that gamma values are lower for Littrow prism spectrographs than for grating and other prism spectrographs. One reason for this is thought to be scattered light reflected from the front surface of the prism and collimating lens of the Littrow instruments to the photographic plate.

### *Speed Ratios:*

The speed ratio data are presented in Tables IV and V. The adjusted averages of the speed ratios are given in Table VI. As stated before, test emulsions on film were compared to SA No. 1 on film while those on plates were compared to SA No. 1 on plates.

Tables IV and V show that the speed ratios were fairly consistent from laboratory to laboratory.

The most obvious conclusion which may be drawn from Table VI is that the SA No. 2 emulsion reacts differently on plates and film. For example, at $D = 0.3$ at 3000 Å, the SA No. 2 plate is only one half as fast as the SA No. 1 plate. The SA No. 2 film, on the other hand, is twice as fast as the SA No. 1 film at that wavelength at the 0.3 density level. The 103-0 emulsion seems to be identical in both plates and film. This emulsion ranges from 3 to 40 times as fast as the SA No. 1 emulsion, depending on the wavelength.

TABLE VII.—PHYSICAL PROPERTIES.

| Emulsion | Chemical Fog | Graininess |
|---|---|---|
| SA No. 1 | 1.00 | 146 |
| SA No. 2 | 1.01 | 157 |
| 103-0 | 0.91 | 201 |
| 33 | 0.93 | 183 |

To make the comparison of the SA No. 2 plate and film more striking it would appear that the plate has only one fourth the speed of the film at $D = 0.3$ and 3000 Å. This would explain many of the inconsistencies in the literature. Many users of film have suggested SA No. 2 as a fast emulsion suitable for trace-element analysis where only tiny samples are on hand, but users of plates of SA No. 2 know it as a slow emulsion not suitable for such uses.

### *Physical Properties:*

In general, there was unanimous agreement that SA No. 1 has superior over-all properties. Everyone noted that the SA No. 2 and 103-0 emulsions are softer and, consequently, have a tendency to distort on rapid drying and to scratch readily. In film, these emulsions curl annoyingly during dry weather and are therefore difficult to use in microphotometry. The backing on 103-0 is quite difficult to remove completely and at best requires the extra processing step of rinsing in dilute sodium carbonate solution.

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[1] R. N. Kniseley and V. A. Fassel, "Effect of Stray Light in Prism and Grating Spectrographs on Emulsion Calibration Curves and Its Significance in Spectrographic Analysis," *Journal*, Optical Soc. of America, Vol. 44, p. 390 (1954).

[2] F. Brech and F. McNally, "Factors Influencing Emulsion Calibration," Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy (March 1953).

[3] Cyrus Feldman, "The Cooperative Standardization of Spectrographic Emulsion Contrast Factors," *Applied Spectroscopy*, Vol. 6, p. 23, Feb. 1952.

Results for chemical fog, defined as the ratio of the clear plate (or film) transmittance after processing to SA No. 1, were quite consistent. These results are averaged in Table VII for plates, but those reported for film are in the same range.

Eastman Kodak provided quantitative information on the graininess of the various plates. Graininess was defined as 1000 divided by the blending magnification. Obviously, as a figure of merit, the smaller the value the finer the grains. Their values are also given in Table VII.

# REPORT OF COMMITTEE E-3
ON
# CHEMICAL ANALYSIS OF METALS*

Committee E-3 on Chemical Analysis of Metals held one meeting during the year: on June 20, 1956, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society. Divisional and other task groups held individual sessions on June 17 to June 20, 1957.

The 1956 edition of the Book of ASTM Methods of Chemical Analysis of Metals is an enlargement of previous editions and represents the endless and fine cooperative effort of many members. It undoubtedly will fill a need in the analytical libraries of those concerned with the ever-changing and complex facets of materials evaluation.

Recognition to Committee E-3 was given by the presentation of the Award of Merit to Harry A. Bright, honorary chairman.

### REVISION OF TENTATIVE

The committee recommends that the Tentative Methods for Chemical Analysis of White Metal Bearing Alloys (**E 57 – 56 T**)[1] be revised by the deletion of Sections 21 and 22 and continued as tentative.

### ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Methods for Chemical Analysis of Antimony Metal (**E 86 – 50 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

### REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

**Standard Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 59 – 45):**[1]

*Section 2.*—Change Paragraphs (*b*) and (*d*) to read as follows:

(*b*) Samples shall consist of drillings or chips cut by some machine tool without the application of water, oil, or other lubricant, and shall be free of scale, surface metal, grease, dirt, or other foreign substances. Except for tubular products, skelp, and flat-rolled stock used in manufacture of pipe and tubes, if samples are taken by drilling, the diameter of the drill used shall be not less than ½ in. when the area of cross-section to be sampled is 16 sq in. and under and shall not be less than 1 in. when the area of cross-section to be sampled is over 16 sq in. In sampling skelp, flat rolled stock used in the manufacture of pipe and tubes, pipe, and tubes the diameter of the drill used shall be the largest practicable within the limits of ¼ to 1 in. Samples shall be uniform, thoroughly mixed, and free of dust. Chips too coarse to pass a No. 16 (1190-micron) sieve are not recommended, nor shall long, curly drillings that will not pack closely for the carbon determination be used. In referring samples to other analysts for check analysis, pieces of the original full-size section, when possible, shall be submitted rather than cuttings, unless the latter are specifically requested.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] 1956 Book of ASTM Methods for Chemical Analysis of Metals.

(*d*) *Bored Forgings, Forged Turned and Bored Pipe.*—For bored forgings, samples shall be taken midway between inner and outer surface of the wall. For forged turned and bored pipe, samples shall be taken by drilling through the pipe wall, cuttings taken across the end of the tube or from broken tensile test specimen.

Add the following new Paragraphs (*i*) and (*j*):

(*i*) *Skelp and Flat Rolled Stock Used In the Production of Pipe and Tubes.*—For skelp and flat rolled stock used in the manufacture of pipe or tubes, the specimen shall be cut across the width of the material as rolled. Sampling shall be by one of the following methods. A specific method may be required by reference.

(*1*) Chips cut by some machine tool representing the full cross-section.

(*2*) Chips cut by some machine tool representing a half cross-section.

(*3*) Drilling through or millings across test specimen or 2 in. strip midway between edge and center.

(*4*) Drilling through at several points across the width.

(*j*) *Pipe or Tubes.*—Sampling shall be by one of the following methods:

(*1*) Chips cut by some machine tool, representing the cross-section.

(*2*) Chips cut by some machine tool, representing a half cross-section located from weld to 180 deg in welded products.

(*3*) Drilling through or cutting by some machine tool across the test specimen or 2-in. coupon located 90 deg to weld in welded products.

(*4*) Drilling through the pipe wall at several locations around the circumference of the pipe or tube.

(*5*) In sampling wrought iron pipe it is desirable that the sample represent the entire cross-section of the specimen. Sampling by milling is preferable.

Complete details are included in Section 6 on sampling wrought iron.

### Photometric Methods for Chemical Analysis of Copper and Copper-Base Alloys (E 62 - 56):[1]

Delete Sections 37 through 43.

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

*Tentative Methods of:*

Chemical Analysis of Ferro-Alloys (E 31 - 54 T),
Methods of Chemical Analysis for Sulfur in Special Brasses and Bronzes (E 54 - 50 T),
Sampling Non-Ferrous Metals and Alloys in Cast Form for Determination of Chemical Composition (E 88 - 50 T),
Chemical Analysis of Copper-Beryllium Alloys (E 106 - 54 T), and

*Tentative Recommended Practice for:*

Photometric Methods for Chemical Analysis of Metals (E 60 - 50 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

## ACTIVITIES OF SUBCOMMITTEES

*Editorial Subcommittee* (J. L. Hague, chairman).—The 1956 Book of ASTM Methods of Chemical Analysis of Metals reflects the efforts of the various members of the Editorial Subcommittee to use a uniform styling in the new material in the book. It is hoped that the remainder of the older material can be processed before the next edition, in order to have a uniform styling of the excellent methods appearing in this text.

Work on the style manual has been largely directed toward accommodating to the Society's style manual sponsored by Subcommittee 15 on Correlation of Chemical Analyses of Committee E-1.

*Division F on Ferrous Metals* (J. J. Furey, chairman) has work in progress on aluminum in steel by a photometric method, cobalt in high-alloy materials by a potentiometric method, hydrogen in steel, with cooperation from Committee A-1 on Steel in setting up a sample procedure, and copper in steel.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Work is also under way on methods for determination of boron, aluminum, silicon, and carbon in ferroboron. This is a joint project with the National Bureau of Standards and includes preparation of a standard sample of ferroboron.

Work is being started on the following new projects: cobalt in stainless steel by a photometric method for type 348 steels with up to 0.20 per cent cobalt; nitrogen in steel by a semi-micro method which may include both soluble and insoluble nitrogen; phosphorus in steel by a photometric method; and columbium, tantalum, titanium, silicon, carbon, tin, and manganese in ferrocolumbium and ferrotantalum-columbium.

*Division G on General Analytical Methods* (H. F. Beeghly, chairman) sponsored a Symposium on Ion Exchange and Chromatography at the 1956 Annual Meeting of the Society and will sponsor a Symposium on Gases in Metals[3] at the 1957 Annual Meeting. These symposia are planned to stimulate the development of improved methods of analysis and the evolution of common analytical procedures for metals.

Increased consideration has been given to methods for establishing the precision and accuracy tolerances for existing analytical methods.

*Division M on Miscellaneous Metals* (R. M. Fowler, chairman), in cooperation with Committee B-9 on Metal Powders, has organized two task groups on the analysis of metal powders. Samples have been exchanged and methods are in preparation for several determinations on copper and tungsten powder.

The cooperative arrangement on methods of analysis for titanium with the Metallurgical Advisory Committee on Titanium of Watertown Arsenal has been continued. Major activity now centers around methods for oxygen in titanium and titanium alloys. Since this subject is of such general interest and importance a paper on this subject has been scheduled for presentation at a symposium at the Annual Meeting.

Future method development projects will include the nickel-molybdenum alloys, electrical heating alloys, and metals of nuclear interest such as zirconium, niobium, and vanadium. Work on analytical methods for these newer metals is contingent on definite and clear requests from the A or B committees concerned.

*Divison N on Non-Ferrous Metals* (R. G. Ernst, chairman) has recommended that all existing tentatives under its jurisdiction be continued without revision, with the exception of Method E 57 – 56 T, which is recommended for revision as stated earlier in the report.

Task groups are studying procedures for the following analyses: determination of antimony with rhodamine-B, copper with neo-cuproine, arsenic by butanol extraction of the arsenomolybdate complex, zinc by means of ethylenediamine tetraacetate (following the extraction of the thiocyanate complex), analysis of fire refined copper, determination of cadmium and zinc in silver solder, magnesium in electronic nickel, and cerium, thorium and rare earths in magnesium base alloys.

A method for the determination of zirconium in magnesium base alloys is nearing completion.

*Division S on Sampling* (A. C. Holler, chairman) appointed a task group to study the sampling of pig iron and the possible revision of Standard Methods of Sampling Steel, Cast Iron, Open Hearth Iron and Wrought Iron (E 59 - 45). It has been recommended that no change be made in the sampling of pig iron. Changes were recommended in the sampling procedures for tubular products for chemical analysis after a cooperative study with Committee A-1 on Steel.

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[3] Issued as a separate publication *ASTM STP No. 222.*

This report has been submitted to letter ballot of the committee, which consists of 128 members; 113 members returned their ballots, of whom 105 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ARBA THOMAS,
*Chairman.*

H. KIRTCHIK,
*Secretary.*

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards revisions of the Tentative Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 - 56 T). The revisions were accepted by the Standards Committee on August 26, 1957 and the revised methods are available as a separate reprint.

# REPORT OF COMMITTEE E-4
# ON
# METALLOGRAPHY*

Committee E-4 on Metallography, together with most of its subcommittees, held two meetings during the year: on June 20, 1956, in Atlantic City, N. J. during the Annual Meeting of the Society, and on February 5, 1957, in Philadelphia, Pa., during ASTM Committee Week.

The committee consists of 113 members.

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued without revision:

*Tentative Methods of*

Preparation of Metallographic Specimens (**E 3 – 46 T**),
Preparation of Micrographs of Metals and Alloys (Including Recommended Practice for Photography as Applied to Metallography) (**E 2 – 49 T**),
Preparing Quantitative Pole Figures of Metals (**E 81 – 54 T**), and
Determining the Orientation of a Metal Crystal (**E 82 – 49 T**).

*Tentative Recommended Practice for:*

Identification of Crystalline Material by the Hanawalt X-Ray Diffraction Method (**E 43 – 49 T**),
Thermal Analysis of Metals and Alloys (**E 14 – 51 T**), and
Dilatometric Analysis of Metallic Material (**E 80 – 49 T**).

## SUBCOMMITTEE ACTIVITIES

*Subcommittee I on Selection and Preparation of Samples* (F. C. Hull, chairman) has in final draft form a revision of the electropolishing section of E 3 – 46 T, Tentative Methods of Preparation of Metallographic Specimens. In this connection, the subcommittee is acting as sponsor for a paper on new electropolishing techniques by the eminent French author M. Jacquet, to be presented at the Annual Meeting.[1]

*Subcommittee III on Nomenclature* (P. A. Beck, chairman) has completed a final draft of the phase nomenclature proposal. This proposal, which offers a new and simplified system of phase designations for alloy systems, will be brought to the attention of other societies and publications in the interest of obtaining a consensus of all metallurgists on this uniform method of phase nomenclature.[2]

*Subcommittee IV on Photography* (S. W. Poole, chairman) through the task group concerned with the photomicrographic exhibit at the Annual Meetings (D. I. Finch, chairman) is making plans for greater student participation in the 1958 showing.

*Subcommittee V on Micro Hardness* (W. A. Shebest, chairman) is engaged in round-robin tests of calibrated test bars and comparators.

*Subcommittee VI on X-ray Methods* (W. L. Fink, chairman) has a task group working on a new and enlarged version of X-ray powder pattern techniques E 43 – 49 T, Tentative Recommended

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] See p. 1290.

[2] ASTM BULLETIN, December, 1957, p. 27 (No. 226).

Practice for Identification of Crystalline Materials by the Hanawalt X-ray Diffraction Method.

*Subcommittee VII on Methods of Thermal Analysis* (D. I. Finch, chairman) plans to review at its June, 1957 meeting a first draft of a method for temperature *versus* electrical resistance measurements.

*Subcommittee VIII on Grain Size* (R. E. Penrod, chairman) has been extremely active in improving the phraseology and clarifying some points in the current E 112 – 55 T, Tentative Methods for Determining the Average Grain Size of Metals. The latest version of this grain-size method has been submitted to Committee B-5 on Copper and Copper Alloys for its consideration with respect to grain counts in copper and copper-base alloys.

Discussions at subcommittee meetings have shown that the expanding technology in materials and techniques apparently necessitate grain-size standards in terms of small-grained structures—for smaller than heretofor considered—and will necessitate the extension of the present system to include such sizes.

*Subcommittee XI on Electron Microstructure of Metals* (S. T. Ross, chairman) has expanded its activities into the nonferrous field by forming a B group to be concerned with titanium alloys and it is planning to form a C group to study the super-strength alloys. Task groups are preparing recommended practices for specimen preparation and for replication techniques for several materials. The Fifth Progress Report on Electron Microstructure of Steel, presented at the 1956 Annual Meeting, is appended to this report.[3] Ths subcommittee is holding a technical session at its June, 1957, meeting, and the papers comprising this session are appended hereto.[4] The First Progress Report of the Non-Ferrous Group was one of these papers.[5]

This report has been submitted to letter ballot of the committee, which consists of 113 members; 79 members returned their ballots, of whom 70 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
*Chairman.*

MARY R. NORTON,
*Secretary.*

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[3] See p. 475.

[4] See pp. 521 through 553.

[5] See p. 454.

## APPENDIX I

# TECHNIQUES EMPLOYED IN THE ELECTRON MICROSCOPE STUDY OF TITANIUM ALLOY WITH 8 PER CENT MANGANESE

## First Progress Report of Non-Ferrous Task Group of Subcommittee XI on Electron Microstructure of Metals, ASTM Committee E-4 on Metallography

At the present time, a need exists for evaluation of methods of specimen preparation and replicating techniques for electron microscopy of non-ferrous materials, similar to that being performed for steels by Subcommittee XI. This need is now being fulfilled by the Non-Ferrous Task Group of Subcommittee XI. Members and participants of this Task Group include:

C. M. Schwartz, *Chairman*—Battelle Memorial Institute.
N. A. Nielsen, *Secretary*—E. I. du Pont de Nemours & Co., Inc.
I. I. Bessen—North American Philips Co., Inc.
L. S. Birks—Naval Research Laboratory.
W. L. Fink—Aluminum Company of America.
W. L. Grube—General Motors Corp.
R. C. Scott—General Motors Corp.
M. S. Hunter—Aluminum Company of America.
J. E. McNutt—E. I. du Pont de Nemours & Co., Inc.
W. Pohl—Ford Motor Co.
R. M. Slepian—Westinghouse Electric Corp.
J. E. Taylor—Chase Brass and Copper Co., Inc.
S. T. Ross—Chrysler Corp.
D. M. Teague—Chrysler Corp.
C. F. Tufts—Sylvania Electric Products, Inc.

So far, work of this body has been directed toward investigating the following aspects of electron metallography of titanium-base alloys:

1. Usefulness of electron microscopy compared to optical microscopy in developing metallographic information.
2. Relative merits of the available techniques of preparing metal specimens for examination and of replicating the specimen.

All research has been performed on specimens from a single heat of a titanium alloy with 8 per cent manganese[1] (C-110M) made by Rem-Cru Titanium, Inc. L. S. Birks arranged for the procurement, heat treatment, and distribution of these specimens. Structures corresponding to nine different heat treatments, described in Table I, were submitted for study by each working member of the committee. Hardness measurements on the specimens were made at the Westinghouse Research Laboratories; these data are also given in Table I.

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[1] Actual chemical analysis: Ingot top: manganese, 7.90; nitrogen$_2$, 0.013; carbon, 0.05; iron, 0.05 per cent. Ingot bottom: manganese, 8.80; nitrogen$_2$, 0.015; carbon, 0.04; iron, 0.03 per cent. Oxygen content: 0.11 per cent. Hydrogen: less than 0.01 per cent.

TABLE I.—STRUCTURES, HEAT TREATMENT, AND HARDNESS OF TITANIUM ALLOY WITH 8 PER CENT MANGANESE.

| Specimen | Heat Treatment | Diamond Pyramid Number, 10 kg | Structure |
|---|---|---|---|
| 1 | 15 min at 1300 F, furnace-cooled[a] to 1050 F, air-cooled | 294 | Massive $\alpha$ in $\beta$ matrix |
| 2 | 15 min at 1300 F, furnace-cooled[a] to 1050 F, air-cooled, reheated to 1300 F for 1 hr, water-quenched | 336 | Massive $\alpha$ in $\beta$ matrix |
| 3 | 1 hr at 1450 F, water-quenched | 372 | Retained $\beta$, with some residual $\alpha$ |
| 4 | 15 min at 1300 F, furnace-cooled[a] to 1050 F, air-cooled, heated to 1450 F, furnace-cooled[a] to 400 F, air-cooled | 287 | Widmanstätten within former $\beta$ grains. Boundaries outlined by continuous $\alpha$ |
| 5 | 1 hr at 1450 F, water-quenched, aged 1 hr at 750 F, air-cooled | 551 | Retained $\beta$, with some residual $\alpha$ |
| 6 | 1 hr at 1450 F, quenched to 750 F, aged 1 hr at 750 F, water-quenched | 533 | Retained $\beta$, with some residual $\alpha$ |
| 7 | 15 min at 1450 F, water-quenched, aged 4 hr at 750 F | 536 | Retained $\beta$, with some residual $\alpha$ |
| 8 | 15 min at 1450 F, water-quenched, aged 4 hr at 850 F | 460 | Retained $\beta$, with some residual $\alpha$ |
| 9 | 15 min at 1450 F, water-quenched, aged 2 hr at 1000 F | 422 | Transformation $\alpha$; some retained $\alpha$ islands |

[a] 5 F per min.

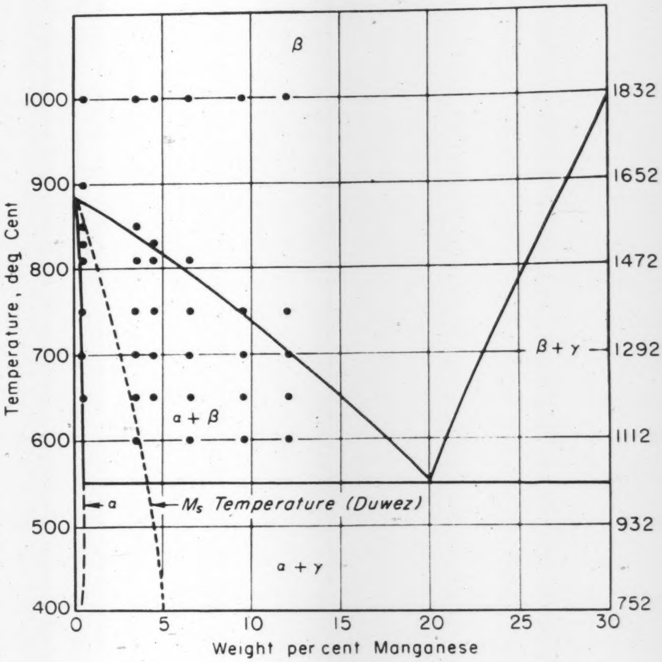

FIG. 1.—Phase Diagram of the Titanium-Manganese System.

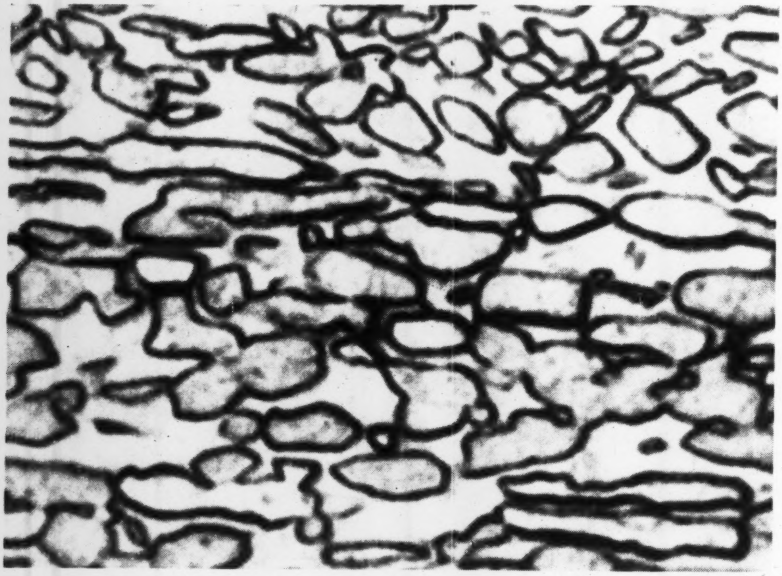

FIG. 2.—Light Micrograph of Specimen No. 1, Mechanically Polished, HF-HNO$_3$ Etch (× 2500).

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

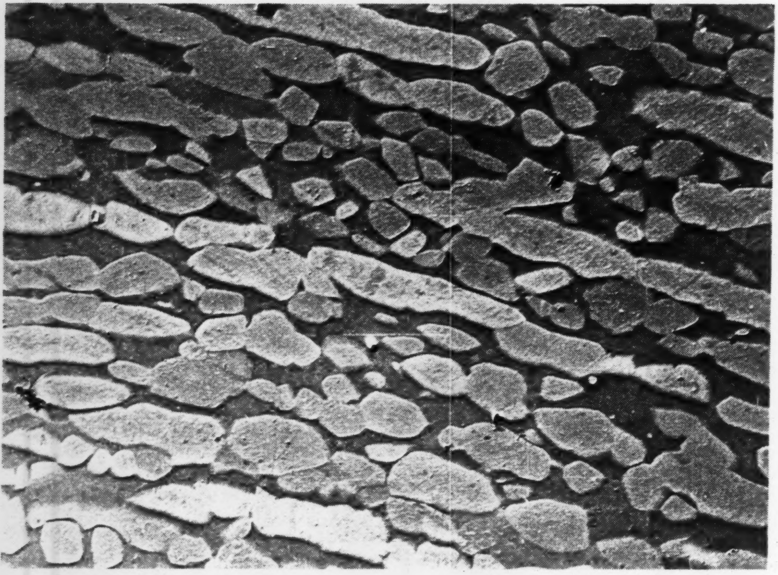

FIG. 3.—Electron Micrograph of Specimen No. 1 Mechanically Polished, HF-HNO$_3$ Etch. Formvar Replica, Chromium Shadowed at 45 deg (× 2500).

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

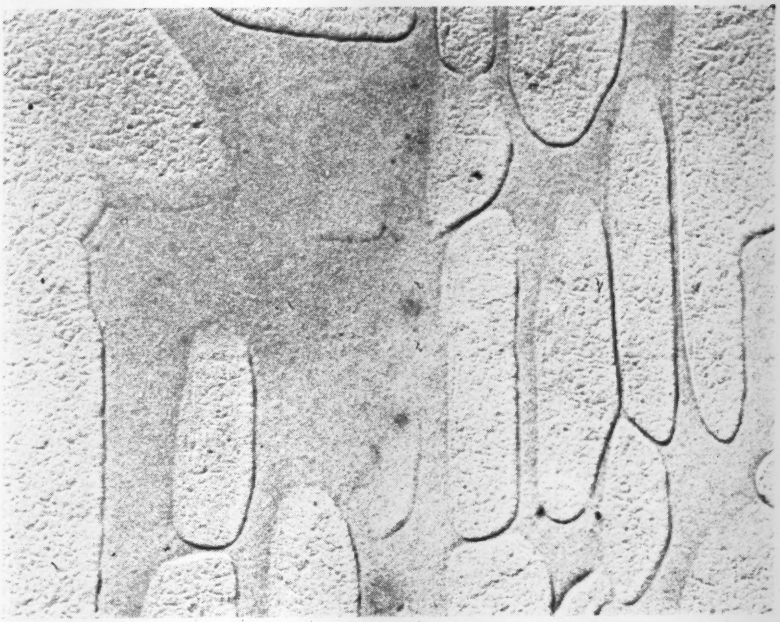

FIG. 4.—Electron Micrograph of Specimen No. 1, Mechanically Polished, HF-HNO$_3$ Etch, Negative Plastic Replica, Shadowed with Platinum (× 15,000).

*Courtesy of Battelle Memorial Institute.*

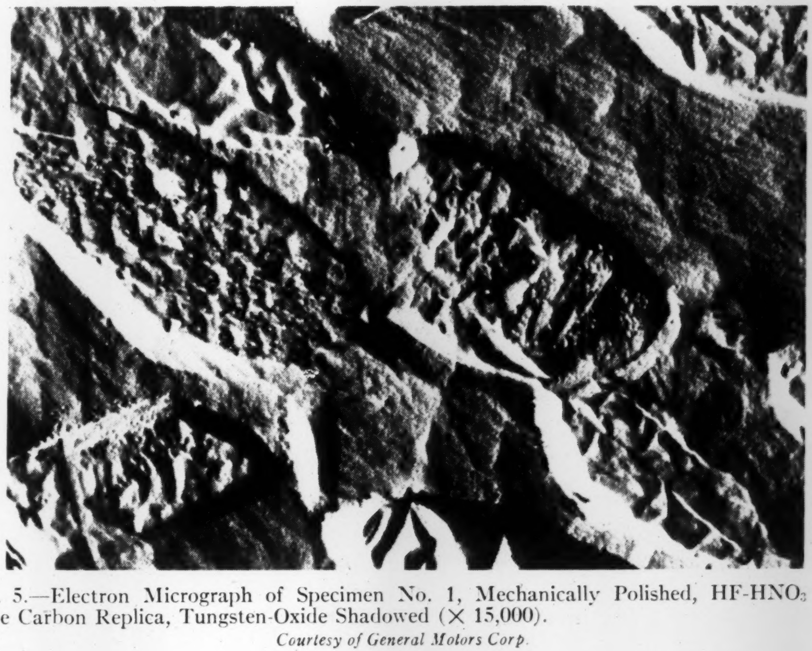

FIG. 5.—Electron Micrograph of Specimen No. 1, Mechanically Polished, HF-HNO$_3$ Etch, Positive Carbon Replica, Tungsten-Oxide Shadowed (× 15,000).

*Courtesy of General Motors Corp.*

## EXPECTED STRUCTURES

Previous studies of the titanium-manganese system (see Fig. 1) made available the following information pertinent to the structures of a titanium alloy with 8 per cent manganese resulting from the heat treatments listed in Table I:

(*a*) At temperatures between approximately 1022 F and 1400 F, a mixture of $\alpha$ and $\beta$ will be the equilibrium structure. Above about 1400 F, the equilibrium condition will be single-phase $\beta$ and below about 1022 F, a mixture of $\alpha$ and $\gamma$.

(*b*) $M_s$ for this alloy is below room temperature (**2**),[2] and, therefore, no martensitic structures should be found.

(*c*) The eutectoid reaction at about 1022 F is sluggish (**1**) and, hence, retained $\beta$ is found in specimens cooled from above the eutectoid temperature.

(*d*) Furnace-cooling from above 1400 F will give grain boundary and Widmanstätten $\alpha$, with retained $\beta$ (**3**).

(*e*) Aging of retained $\beta$ by precipitation of $\alpha$ occurs at temperatures below the eutectoid, but no precipitates are found by light microscopy until overaging occurs (**3**). $\alpha$-particle size increases with increasing aging temperature. X-ray diffraction studies show that $\omega$ may appear as a transition phase before $\alpha$ is formed (**4**).

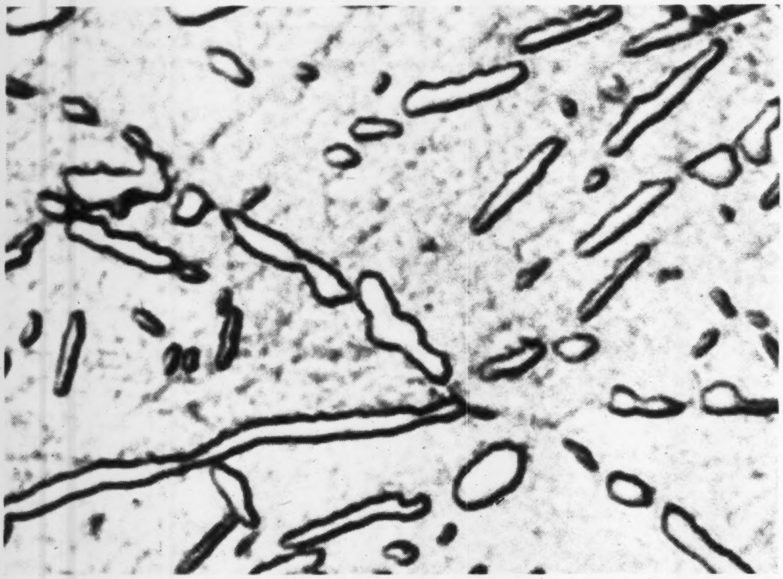

FIG. 6.—Light Micrograph of Specimen No. 2, Mechanically Polished, HF-HNO$_3$ Etch (× 2500).
*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

## METALLOGRAPHY

Contributions from various members of the group permitted comparisons of electron micrographs representing various polishing and etching techniques and replicating methods, which are reviewed in the Appendix to this report.[3] Optical photomicrographs are included and discussed in the report as an aid in

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[2] The boldface numbers in parentheses refer to the list of references appended to this report, see p. 472.

[3] See p. 473.

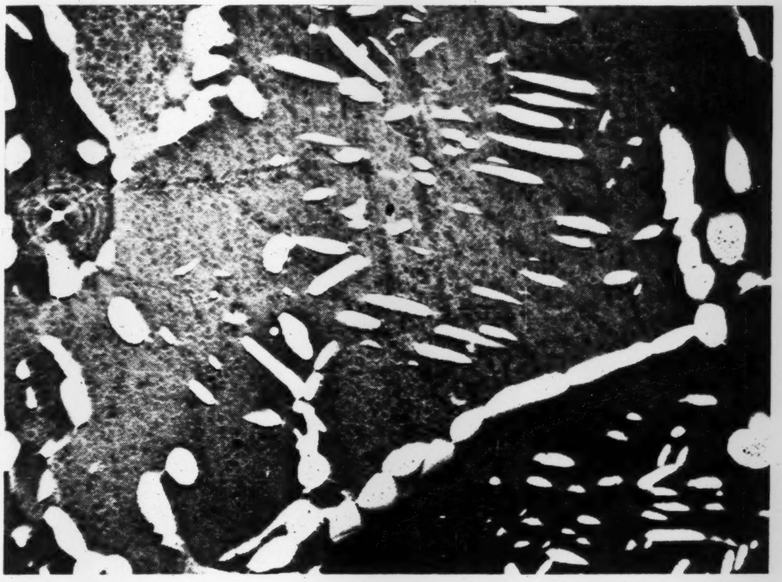

FIG. 7.—Electron Micrograph of Specimen No. 2, Mechanically Polished, HF-$HNO_3$ Etch, Formvar Replica, Chromium Shadowed at 45 deg (× 2500).

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

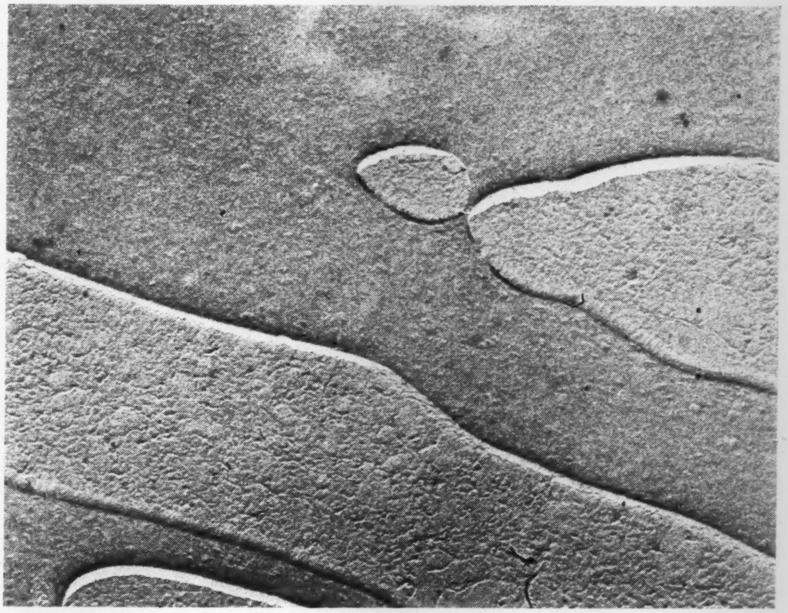

FIG. 8.—Electron Micrograph of Specimen No. 2, Mechanically Polished, HF-$HNO_3$ Etch, Negative Plastic Replica, Shadowed With Platinum at 45 deg (× 15,000).

*Courtesy of Battelle Memorial Institute.*

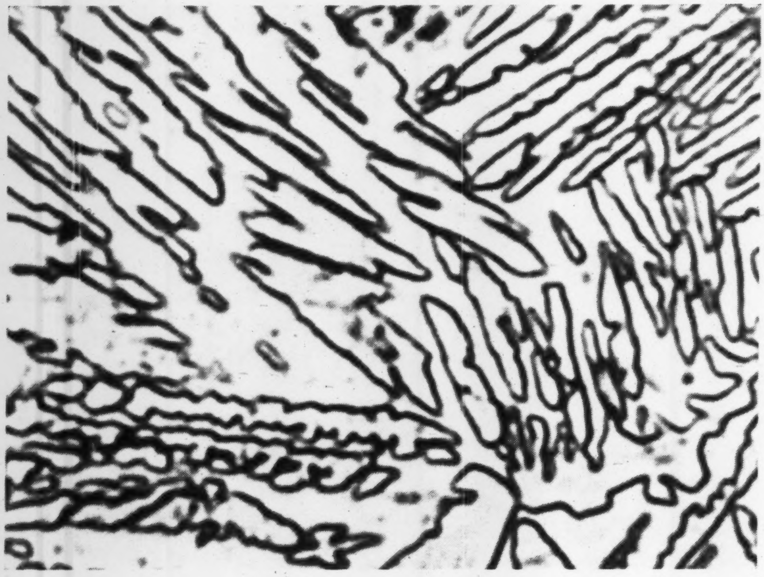

FIG. 9.—Light Micrograph of Specimen No. 4, Mechanically Polished, HF-HNO$_3$ Etch (× 2500)

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

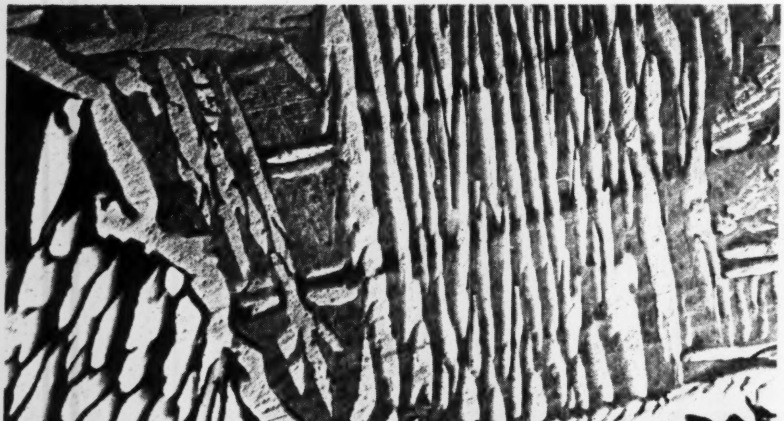

FIG. 10.—Electron Micrograph of Specimen No. 4, Mechanically Polished, HF-HNO$_3$ Etch. Formvar Replica, Chromium Shadowed at 45 deg (× 2500).

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

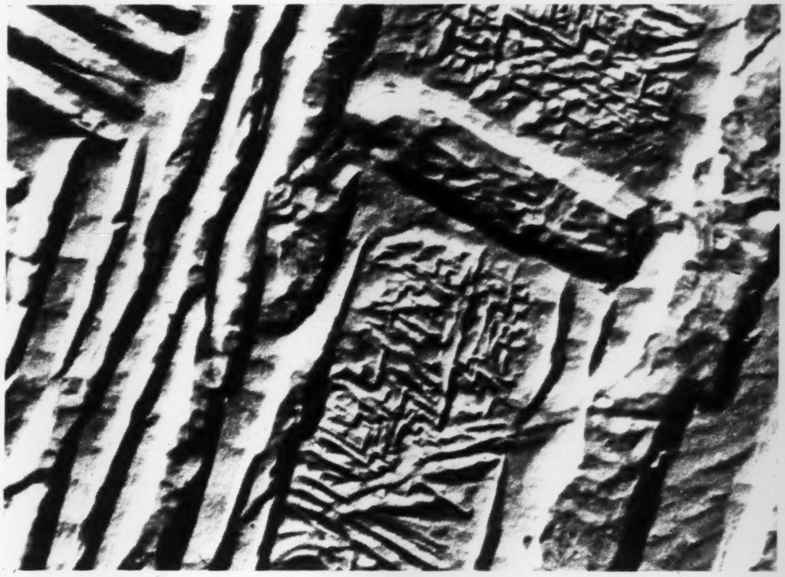

FIG. 11.—Electron Micrograph of Specimen No. 4, Mechanically Polished, HF-HNO$_3$ Etch, Positive Carbon Replica (× 15,000).

*Courtesy of General Motors Corp.*

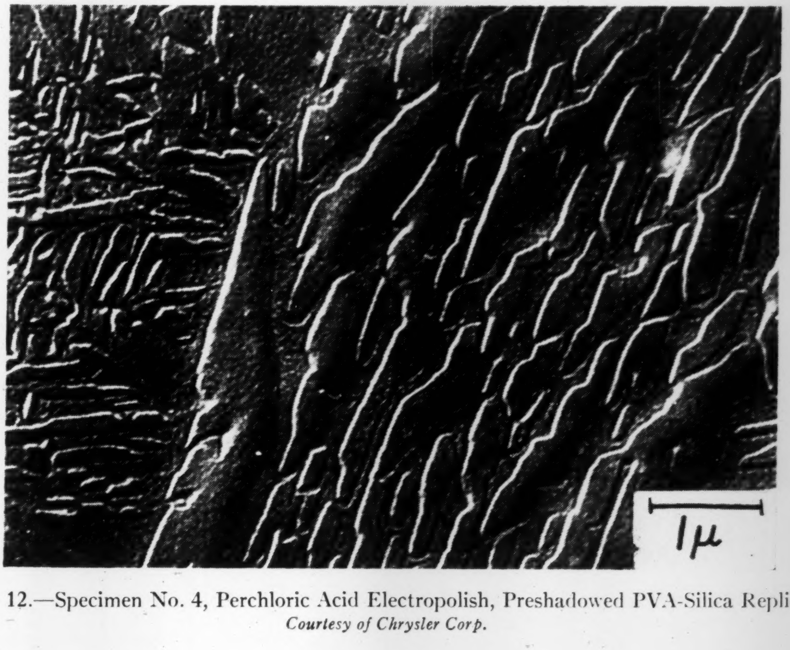

FIG. 12.—Specimen No. 4, Perchloric Acid Electropolish, Preshadowed PVA-Silica Replica.

*Courtesy of Chrysler Corp.*

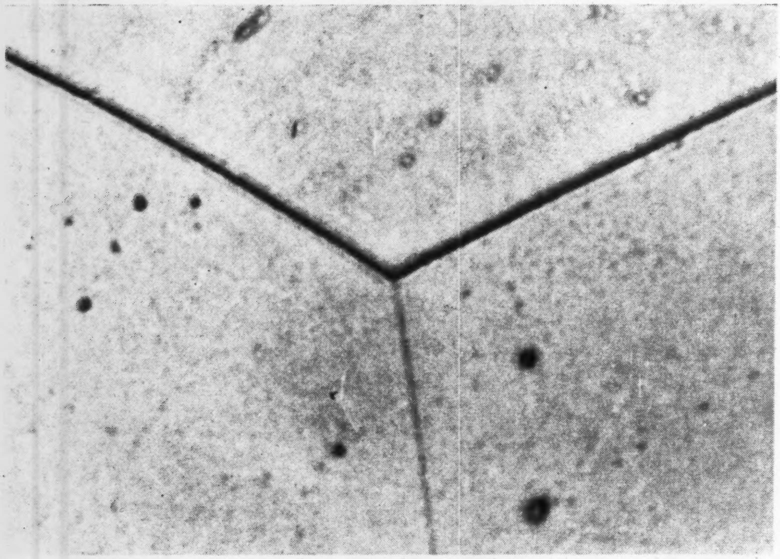

FIG. 13.—Light Micrograph of Specimen No. 3, Mechanically Polished, HF-$HNO_3$ Etch (× 2500).
*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

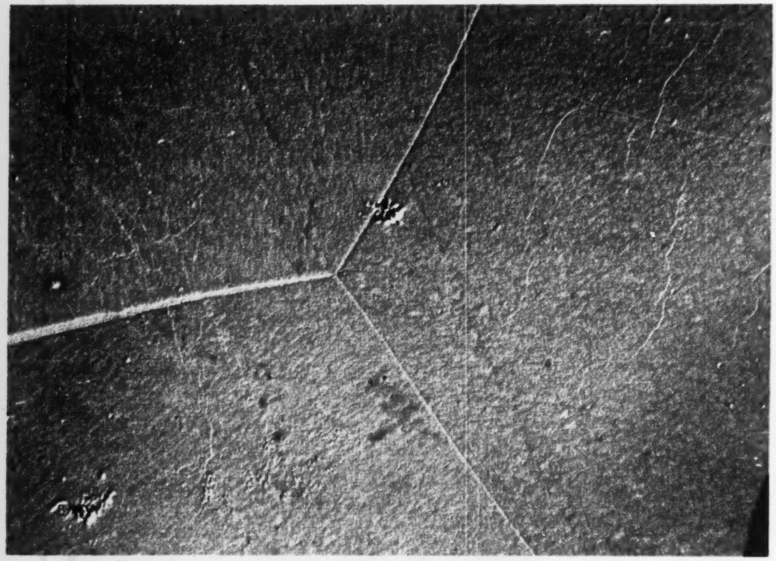

FIG. 14.—Electron Micrograph of Specimen No. 3, Mechanically Polished, HF-$HNO_3$ Etch, Formvar Replica, Chromium Shadowed at 45 deg (× 2500).
*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

evaluating circumstances where the superior resolution obtainable by electron microscopy is advantageous in metallographic studies.

Specimen No. 1 (Figs. 2 to 5) was intended to approximate the alloy condition after hot rolling. Specimen No. 2 (Figs. 6 to 8) was prepared for comparison with No. 1, to see if differences in the retained $\beta$ phase could be indicate whether there is $\alpha$ decomposition.

There is also a small difference in the structure of the $\beta$ in specimens Nos. 1 and 2, as evidenced by a faint nodular appearance of the matrix in Figs. 4 and 8. This fine structure was not evident in micrographs from other laboratories, presumably because of heavier etching. The specimens of Figs. 4 and 8 were polished and etched together in one mount, so the difference in $\beta$ structure probably denotes a difference in the nature of the $\beta$ in the two specimens. According to the phase diagram, the $\beta$ in specimen No. 2 would contain about 12 per cent manganese. Previous experience has indicated that $\beta$ of this composition is stable on quenching. However, it is possible to detect decomposition in the initial stage by electron microscopy before it can be detected by either X-ray diffraction or light microscopy.

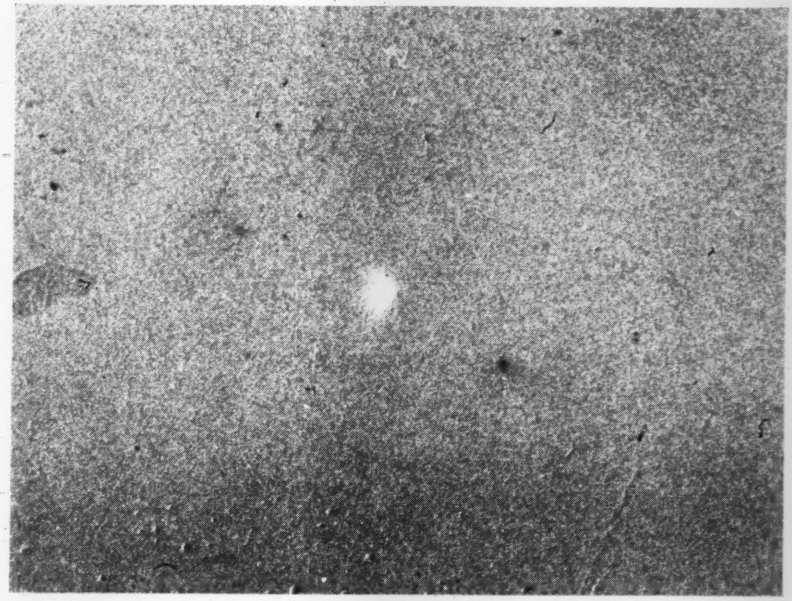

FIG. 15.—Electron Micrograph of Specimen No. 3, Mechanically Polished, HF-HNO$_3$ Etch Positive Silica Replica, Preshadowed With Platinum at 45 deg (× 25,000).

*Courtesy of Battelle Memorial Institute.*

detected; presumably the $\beta$ in No. 1 would be more highly supersaturated, since less was present. There is a fine threadlike structure in the $\alpha$ grains in specimen Nos. 1 and 2 as shown in Figs 4. and 8. This could be due to decomposition of $\alpha$ on cooling below the eutectoid temperature. The significance of this structure could probably be better ascertained in an alloy of low manganese content. The effect of solution treating temperature and cooling rate on the hardness of the $\alpha$ would

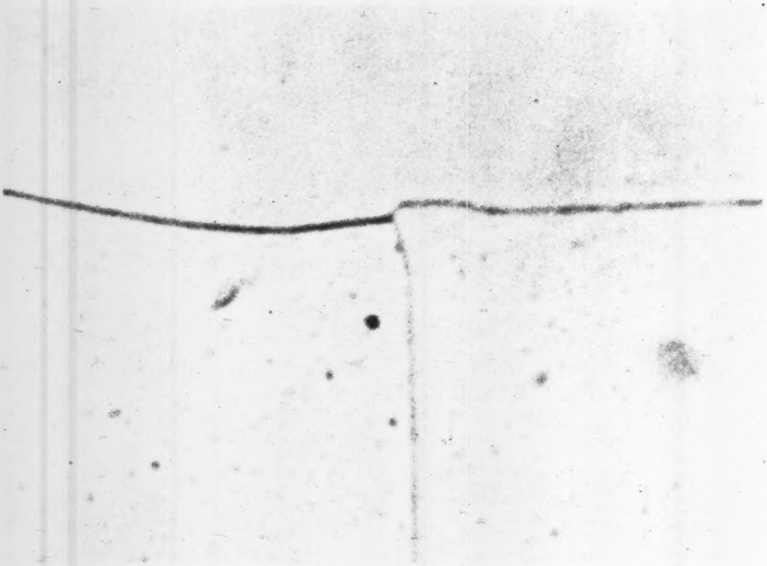

FIG. 16.—Light Micrograph of Specimen No. 5, Mechanically Polished, HF-HNO$_3$ Etch (× 2500).

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

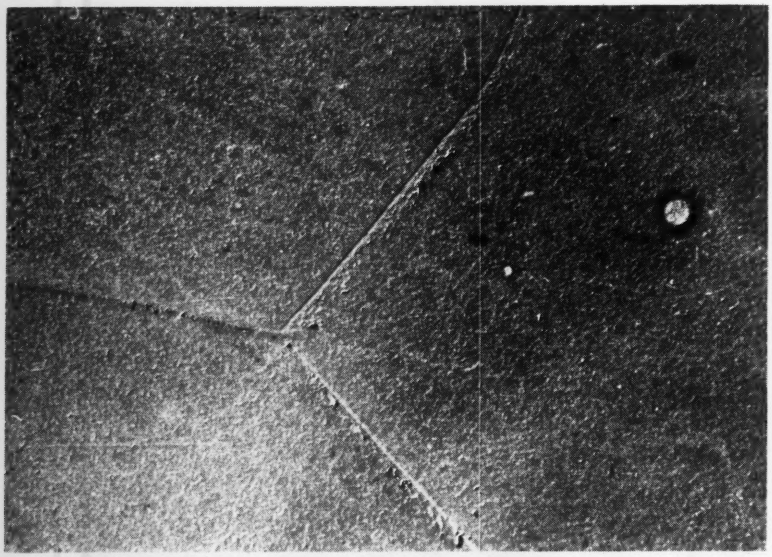

FIG. 17.—Electron Micrograph of Specimen No. 5, Mechanically Polished, HF-HNO$_3$ Etch, Formvar Replica, Chromium Shadowed at 45 deg (× 2500).

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

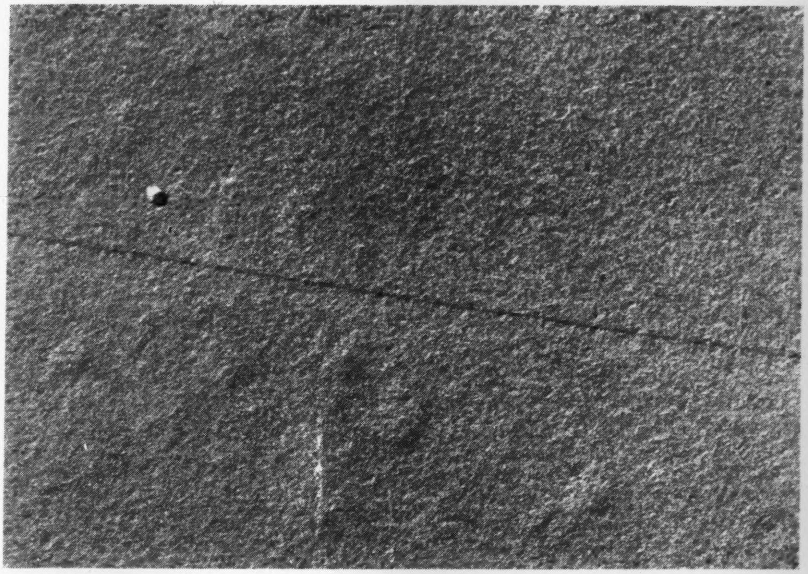

FIG. 18.—Electron Micrograph of Specimen No. 5, Mechanically Polished, HF-$HNO_3$ Etch Positive Silica Replica, Preshadowed with Platinum at 45 deg (× 25,000).

*Courtesy of Battelle Memorial Institute.*

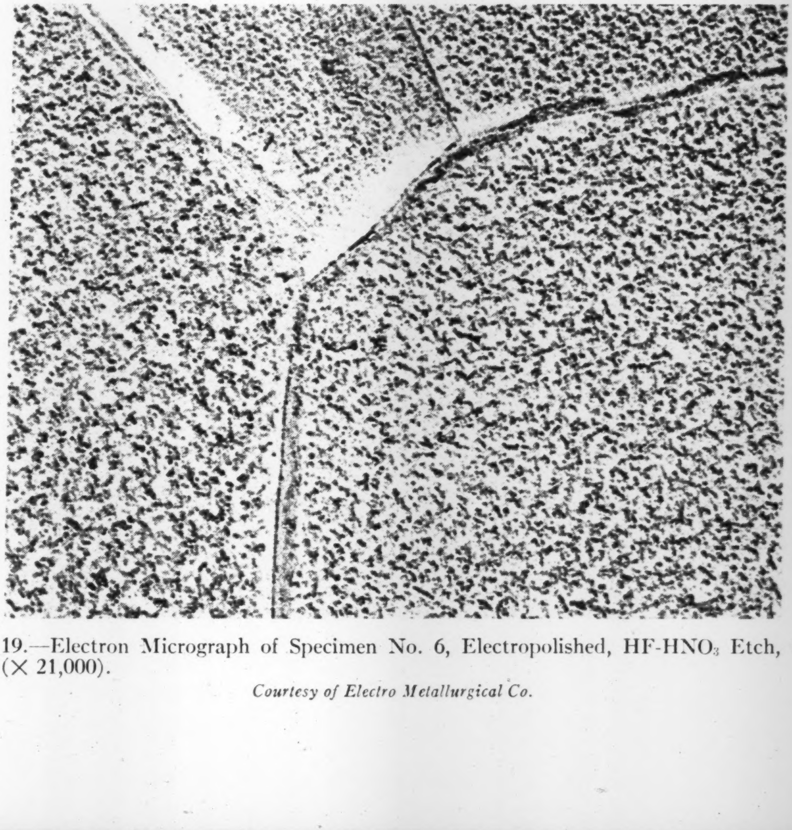

FIG. 19.—Electron Micrograph of Specimen No. 6, Electropolished, HF-$HNO_3$ Etch, Carbon Replica (× 21,000).

*Courtesy of Electro Metallurgical Co.*

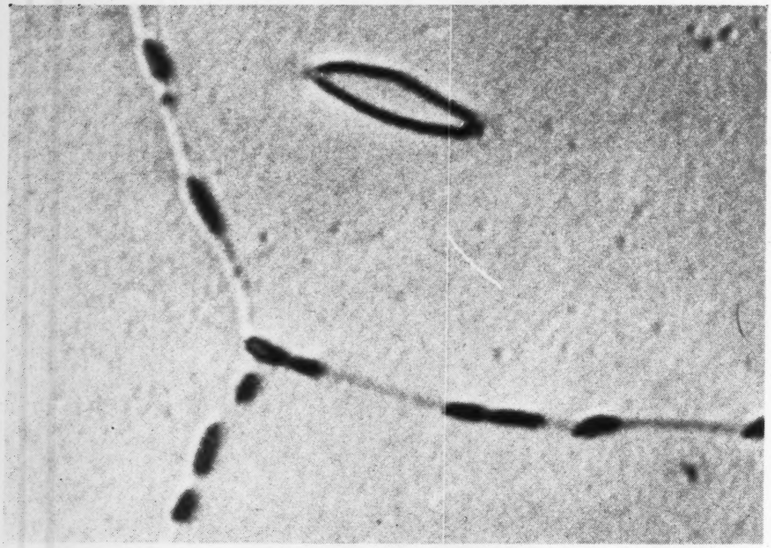

FIG. 20.—Light Micrograph of Specimen No. 7, Mechanically Polished, HF-HNO$_3$ Etch (× 3000).
*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

Therefore, it is suggested that specimen 2 has undergone initial decomposition of the $\beta$, whereas specimen No. 1, with higher manganese content in the $\beta$, is more stable.

Figure 5 shows specimen No. 1 after heavy etching. A subgrain structure is observed in both phases after overetching. Figures 3 and 7 show some difference in the distribution of $\alpha$ in specimens Nos. 1 and 2. It is interesting that the $\alpha$ remaining in specimen No. 2 is sometimes nearly continuous at $\beta$ grain boundaries and that within the grain, a crude orientation relation often remains.

It was the consensus that electron microscopy revealed textural differences of uncertain significance between the $\alpha$ and $\beta$ phases and that, other than this, there was little in these specimens that could not be seen with optical microscopy. Negative plastic replicas were as satisfactory as more elaborately prepared replicas.

Specimen No. 4 (Figs. 9 to 12) was heat treated to develop the structure of No. 1, then reheated to 1450 F and furnace-cooled to 400 F. Comparison of light and electron micrographs shows superior resolution of the fine Widmanstätten structure in the latter. In some

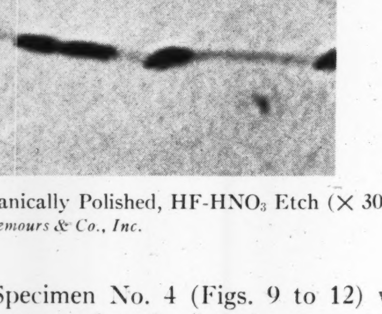

FIG. 21.—Electron Micrograph of Specimen No. 7, Electropolished, HF-HNO$_3$ Etch, Collodion Replica, Manganese Shadowed at 15 deg (× 3000).
*Courtesy of Naval Research Lab.*

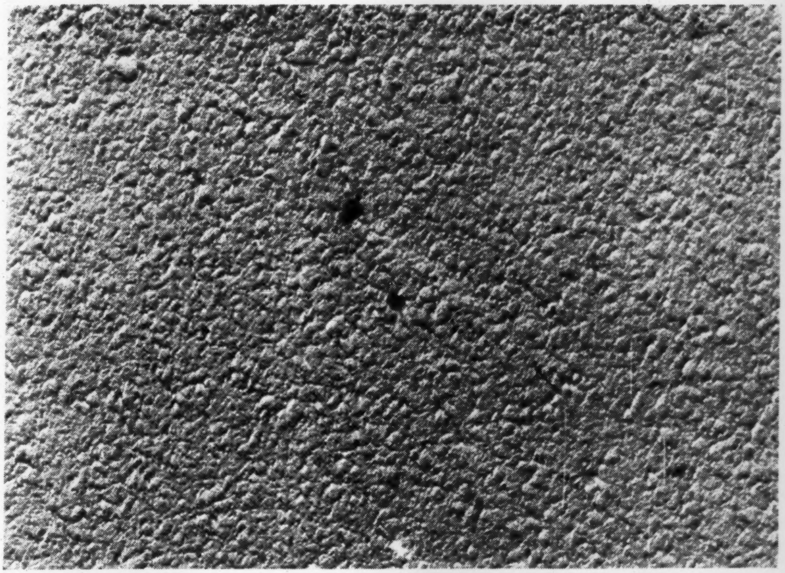

FIG. 22.—Electron Micrograph of Specimen No. 7, Mechanically Polished, HF-$HNO_3$ Etch, Positive Silica Replica, Preshadowed with Platinum (× 25,000).

*Courtesy of Battelle Memorial Institute.*

grains, α plates having the Widmanstätten orientation grow outward from the α nearly continuous along former β grain boundaries. Similar structures are often found in proeutectoid ferrite forming from austenite in iron-carbon alloys. However, a question remains as to whether the α along the former β grain boundaries remained after heat treatment in the β region or if it precipitated upon cooling below the β transus. Undissolved α remains in specimens Nos. 7, 8, and 9, which spent approximately the same time in the temperature range where only β is stable, so residual α might also be expected in specimen No. 4. Longer homogenizing treatments would have been desirable to dispel this question and also for other reasons, as will be discussed below.

For specimen No. 4, plastic replicas were found suitable for metallographic studies.

Specimens Nos. 3, 5, 6, and 7 were intended to be a series to show the progress of precipitation during aging of retained β at 750 F. Suitability of these specimens for that purpose may be questioned, on the basis of inadequacy of the homogenizing heat treatment. Banding is evident, leading to zones where α still remained after 1 hr at 1450 F; the effect is most pronounced in specimen No. 7, which had a shorter homogenizing treatment. This necessitated special care by members of the Task Group, to assure that they were comparing regions equivalent in manganese content, since variations in alloy concentration can be expected to have significant effects on the nature and kinetics of precipitation. X-ray diffraction analyses of specimens Nos. 5 and 7 performed at Battelle Memorial Inst. showed that both specimens contained ω and enriched β. Specimen No. 7 contained more ω than specimen 5.

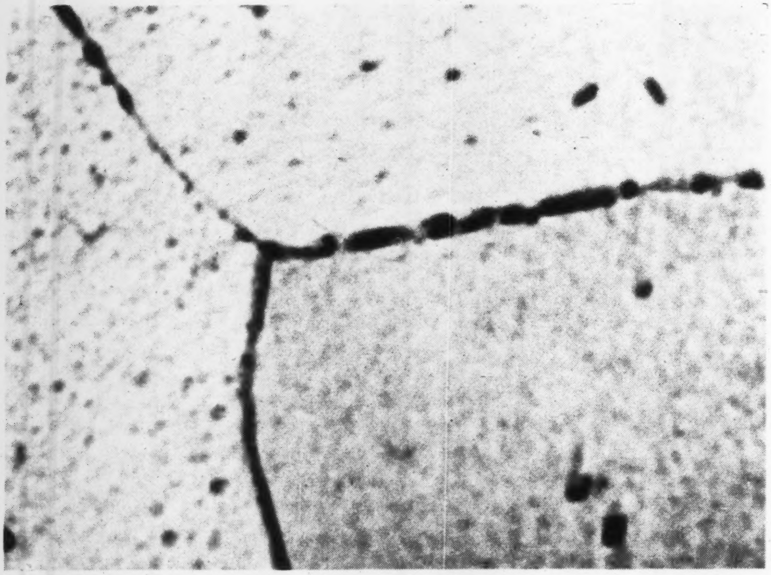

FIG. 23.—Light Micrograph of Specimen No. 8, Mechanically Polished, HF-HNO$_3$ Etch (× 3000.)
*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

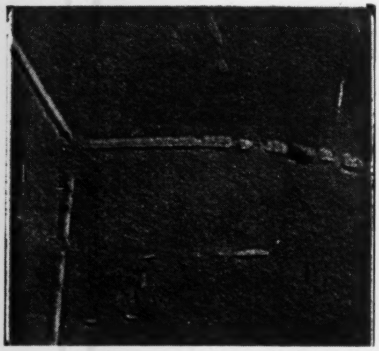

FIG. 24.—Electron Micrograph of Specimen No. 8, Electrolytically Polished, Electrolytic HF-HNO$_3$ Etch, Collodion Replica, Manganese Shadowed at 15 deg (× 3000).
*Courtesy of Naval Research Lab.*

Table I shows that specimens Nos. 5, 6, and 7 all have the characteristic high hardness of titanium alloys with an appreciable $\omega$ content.

Specimen No. 3 (Figs. 13 to 15) exhibits retained $\beta$, though as mentioned above, regions in which $\alpha$ exists can be found. By prolonged etching with dilute aqueous HF-HNO$_3$, an array of apparently crystallographic pits suggestive of a subgrain structure can be developed in many $\beta$ grains, but this is not observed in replicas suitable for electron microscopy.

Specimens Nos. 5 (Figs. 16 to 18) and 6 (Fig. 19) were aged 1 hr at 750 F. They were quenched according to different procedures. The precipitation due to aging would be expected to be more pronounced in No. 6 than in No. 5, because of the slower quenching rate above the aging temperature. Comparison of Figs. 18 and 19 indicates that this is the case. Precipitation of the $\omega$ phase is more pronounced in specimen No. 6. However, in this comparsion the difference in specimen preparation must be considered. Specimen No. 5 was mechanically polished before etching; specimen No. 6 was electropolished. The precipitate in specimen No. 6 is similar in appearance to that in a

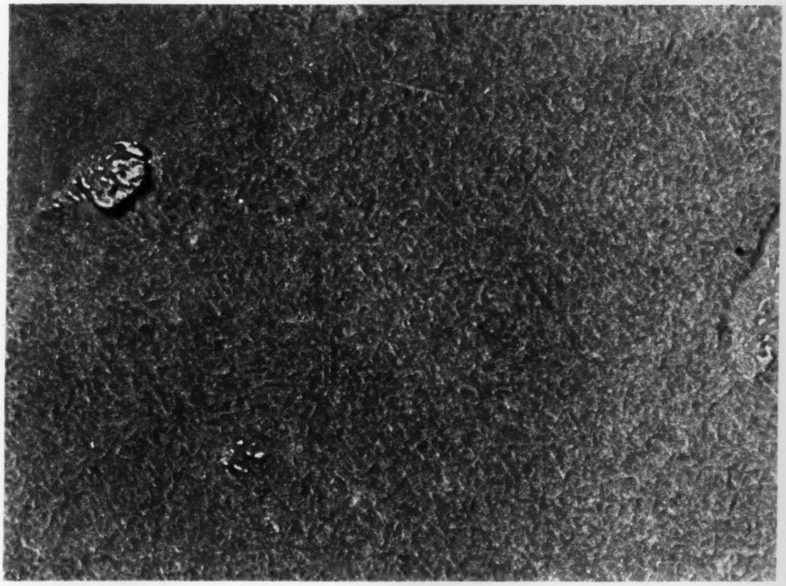

FIG. 25.—Electron Micrograph of Specimen No. 8, Electrolytically Polished, HF-$HNO_3$ Etch, Collodion Replica, Manganese Shadowed at 15 deg (× 15,000).

*Courtesy of Naval Research Lab.*

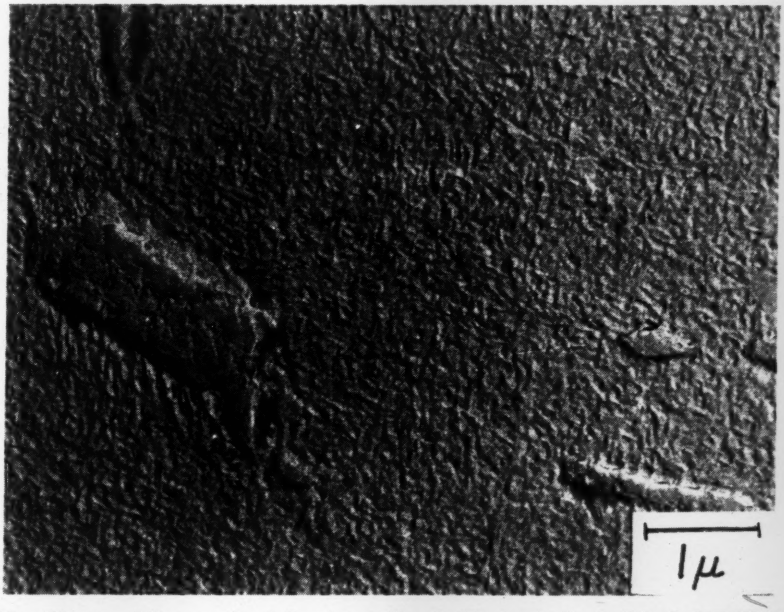

FIG. 26.—Electron Micrograph of Specimen No. 8, Mechanically Polished, HF-$HNO_3$ Etch, Two-Stage Carbon Replica (× 15,000).

*Courtesy of Westinghouse Electric Corp.*

specimen of an ω bearing titanium with 8 per cent chromium beta-quenched and aged, which was electropolished before etching (**5**). Apparently, electropolishing should be used in preference to mechanical polishing as a pretreatment before etching, in an electron microscope investigation of β aging.

The roughening is progressive as the degree of aging increases, as shown by examination of specimen No. 7 (Figs. 20 to 22) and No. 8 (Figs. 23 to 26). This roughening is on a scale too fine to be resolved by light microscopy. The precipitate in specimen No. 8 is probably α, as suggested by the reduced hardness (**4**). It is interesting to note that the α is acicular in contrast to nodular ω, in specimens Nos. 5, 6, and 7. ω appears to be cubic in structure with a large cell, isomorphous with a multiple of the cubic cell in the β (**6**). There is then no preferred orientation for the ω precipitate whereas the α will emerge in platelets of specific orientations, to minimize the surface energy. In the aging series (specimens Nos. 3, 5, 7, and 8), plastic replicas were inadequate for resolving the fine degree of roughening progressively developed. Both direct carbon and the metal-silica replicas (the latter represented by the Battelle Memorial Inst. contribution to this report) were noticeably superior. It is believed that replica thickness relative to the thickness of shadow-cast metal controls resolving power in these replicas.

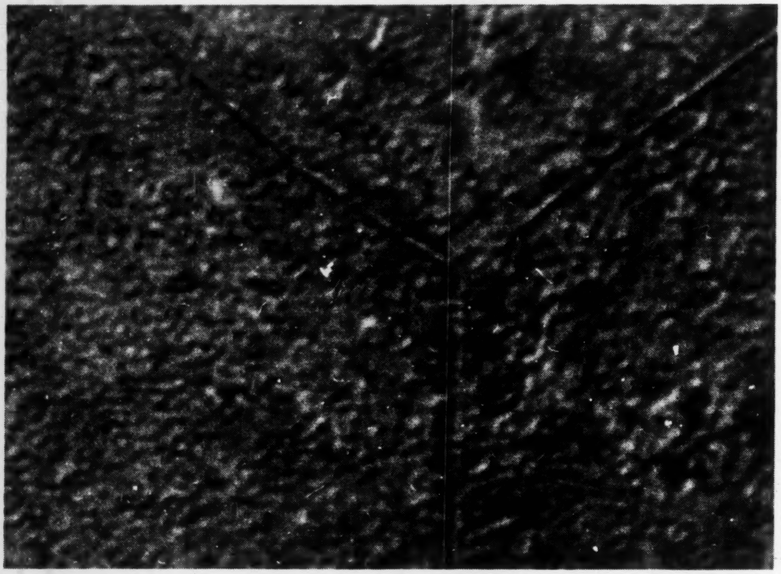

FIG. 27.—Light Micrograph of Specimen No. 9, Mechanically Polished, HF-HNO$_3$ Etch (× 2500)

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

Specimen No. 9 (Figs. 27 to 29) represents an advanced stage in the precipitation of α from supersaturated β below the eutectoid temperature. The α phase is acicular, occurring as closely spaced plates along grain boundaries

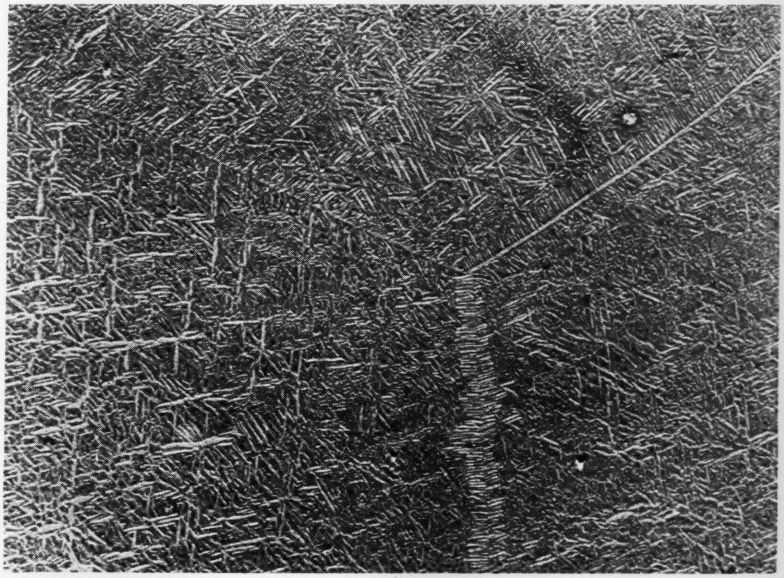

FIG. 28.—Electron Micrograph of Specimen No. 9, Mechanically Polished, HF-HNO$_3$ Etch, Formvar Replica, Chromium Shadowed at 45 deg (× 2500).

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

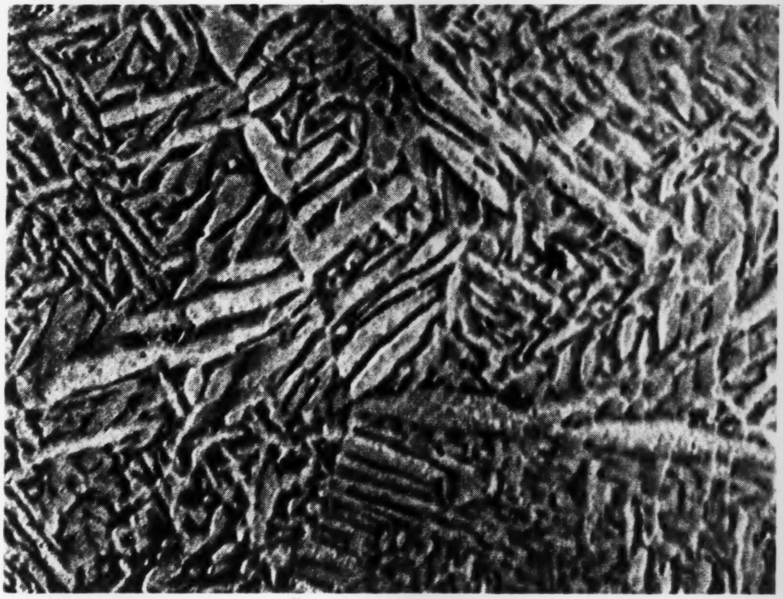

FIG. 29.—Electron Micrograph of Specimen No. 9, Mechanically Polished, HF-HNO$_3$ Etch, Formvar Replica, Chromium Shadowed at 45 deg (× 15,000).

*Courtesy of E. I. du Pont de Nemours & Co., Inc.*

and as spherulitic structures within the grains. Here the superior resolution of structure by electron microscopy is apparent, and plastic replicas seem as well suited for examination as other kinds.

In summary, electron microscopy appears to be advantageous in examining the following structures in a titanium alloy with 8 per cent manganese:

1. Roughening of retained $\beta$ during subcritical aging but prior to formation of resolvable $\alpha$ or $\omega$ particles.

2. Fine Widmanstätten $\alpha$ in $\beta$ aged at high subcritical temperatures.

3. Substructures in $\alpha$ and $\beta$ phases developed by prolonged etching attack.

## REFERENCES

(1) D. J. Maykuth, H. R. Ogden, and R. I. Jaffee, "Titanium-Manganese System," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 197, pp. 225-30 (1953).

(2) P. Duwez, "The Martensite Transformation Temperature in Titanium Binary Alloys," *Transactions*, Am. Soc. Metals, Vol. 45, pp. 934-40 (1953).

(3) F. C. Holden, H. R. Ogden, and R. I. Jaffee, "Heat Treatment, Structure, and Mechanical Properties of Titanium-Manganese Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 200, pp. 169-84 (1954).

(4) P. D. Frost, W. M. Parris, L. L. Hirsch, J. R. Doig, and C. M. Schwartz, "Isothermal Transformation of Titanium-Manganese Alloys," *Transactions*, Am. Soc. Metals, Vol. 46, pp. 1056-74 (1954).

(5) P. D. Frost, W. M. Parris, L. L. Hirsch, J. R. Doig, and C. M. Schwartz, "Isothermal Transformation of Titanium-Chromium Alloys," *Transactions*, Am. Soc. Metals, Vol. 46, pp. 231-256 (1954).

(6) A. E. Austin, and J. R. Doig, "Structure of the Transition Phase Omega in Titanium-Chromium Alloys," *Journal of Metals*, Vol. 9, Jan., 1957, pp. 27-30.

## APPENDIX

## SUMMARY OF TECHNIQUES EMPLOYED IN THE ELECTRON MICROSCOPE STUDY OF TITANIUM ALLOY WITH 8 PER CENT MANGANESE

The following is a brief summary of the methods used by members of the Task Group in obtaining the electron micrographs described in the report. No attempt is made at this time to decide which technique is best for a particular structure.

### SAMPLE PREPARATION

*Mechanical Polishing:*

The procedures employed were essentially the same as those used for light microscopy. Alternate polishing and etching or use of dilute aqueous HF to wet the final wheel is essential to minimize disturbed metal on the surface.

*Electropolishing:*

Electrolytic polishing was performed using the proprietary[4] perchloric-acid-base A-3 electrolyte, after hand polishing through 000 paper.

*Chemical Polishing:*

A patented[5] solution of the following composition was used for chemical polishing: 200 ml 70 per cent $HNO_3$, 125 ml 30 per cent $H_2SiF_6$, 30 g $NH_4HF_2$. Specimens were previously rough ground through 0000 paper and were then immersed in the polishing solution long enough to remove scratches.

### ETCHANTS

Some specimens were replicated without intentional etching after electropolishing or chemical polishing. Etching was performed either by simple immersion or anodic dissolution in an aqueous solution of 2 per cent HF plus 4 per cent $HNO_3$.

### REPLICAS

*Negative Plastic Replicas:*

Both dry-stripped collodion and wet-stripped, collodion-backed Formvar replicas were used. In the latter, backing and stripping under water are frequently used to minimize strain. Shadowing is required to develop contrast.[6]

*Negative Carbon Replicas:*

Thin films of carbon evaporated normally onto prepared specimen surfaces and freed by immersion in the metallographic etchant were used for studying some specimens. Shadowing was used to enhance contrast.

*Positive Carbon Replicas:*

Specimen surfaces were covered with a 4 per cent solution of collodion in amyl acetate, then allowed to drain at 45 deg to the horizontal and dry. Before drying, 200-mesh copper screens were imbedded in the collodion. After drying, the films were dry-stripped with masking tape; then a shadowing material and carbon were evaporated onto the replica side of the films. The composites were then placed in a closed beaker of amyl acetate so that solvent washed from the underside of the screen and dissolved the collodion, after which the carbon films were ready for examination.

*Preshadowed Silica Replicas:*

(*a*) A thick first replica was made of polyvinyl alcohol and shadowed with chromium at an angle of 30 deg from the plane of the replica. The final replica was prepared by depositing a thin film of silica normally to the shadowed polyvinyl acetate (PVA) surface. Separation of the PVA and silica was accomplished by floating the composite film on the surface of warm water which dissolved the PVA.

---

[4] Uddeholm Co. of America, Inc.

[5] U. S. Patent No. 2,711,364.

[6] Additional data on replication procedures are contained in: Symposium on Techniques for Electron Metallography, Am. Soc. Testing Mats (1953). (Issued as separate publication *ASTM STP No. 155*); "Electron Microstructure of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 444 (1950).

(*b*) Cellulose acetate tape (formerly marketed as Faxfilm) moistened in acetone was pressed against the specimen surfaces and allowed to dry. After dry-stripping, platinum was evaporated, at a 1 to 1 shadow angle, on the replica side. Silica was then evaporated at normal incidence. The composite replica was cut into $\frac{1}{8}$-in. squares and the cellulose acetate tape dissolved away in acetone.

## APPENDIX II

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# ELECTRON MICROSTRUCTURE OF BAINITE, PEARLITE, AND TEMPERED MARTENSITE IN AISI 4140 STEEL

## FIFTH PROGRESS REPORT BY SUBCOMMITTEE XI OF COMMITTEE E-4*

Since its formation in 1948, Subcommittee XI on the Electron Microstructure of Metals has investigated the structures produced by heat treatment of an eutectoid carbon steel and revealed by electron microscopy. The cooperative investigation included electron metallographic examination of isothermal transformation structures of austenite ranging from coarse pearlite (1300 F) to lower bainite (500 F), and tempered martensite and tempered bainite. The electron micrographs obtained have been published and discussed in detail in four progress reports by the subcommittee in the Society's *Proceedings* for 1950, 1952, 1953, and 1954 (**1–4**).[1] Subsequent to these reports, the results of some further studies on this steel were presented to the Society during the 1955 Annual Meeting by individual members of Subcommittee XI.

To extend the usefulness of the detailed investigation of eutectoid carbon steel, the subcommittee undertook a brief survey of the electron microstructures of a steel representative of the medium alloy, medium carbon group of heat-treatable steels that are of considerable commercial interest. An AISI type 4140 steel was chosen as typical of such steels, and a series of specimens was prepared, including quenched-and-tempered structures as well as structures resulting from both partial and complete isothermal transformation. These specimens were examined, using essentially the same electron metallographic techniques as in the previous eutectoid carbon steel program. While the results reported below are similar to those obtained on the other steel, some important differences do exist.

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* Members of Subcommittee XI actively participating in this study:

S. T. Ross, Chrysler Corp., Detroit, Mich., *Chairman* 1956–1958.

R. M. Fisher, U. S. Steel Corp., Monroeville, Pa., *Chairman* 1954–1956.

I. I. Bessen, North American Philips Co., Mt. Vernon, N. Y.

L. S. Birks, Naval Research Laboratories, Washington, D. C.

A. L. Ellis, International Harvester Co., Chicago, Ill.

W. L. Grube, General Motors Corp., Detroit, Mich.

E. J. Korda, Curtiss-Wright Corp., Wood Ridge, N. J.

G. E. Noakes, Ford Motor Co., Dearborn Mich.

L. Pellier, American Cyanamid Co., Stamford, Conn.

G. E. Pellissier, U. S. Steel Corp., Monroeville, Pa.

G. H. Robinson, General Motors Corp., Detroit, Mich.

F. G. Rowe, American Cyanamid Co., Stamford, Conn.

C. M. Schwartz, Battelle Memorial Institute, Columbus, Ohio.

D. M. Teague, Chrysler Corp., Detroit, Mich.

C. F. Tufts, Sylvania Electric Products, Bayside, L. I., N. Y.

J. R. Vilella, U. S. Steel Corp., Monroeville, Pa.

[1] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 520.

TABLE I.—STRUCTURES INVESTIGATED AND CORRESPONDING HEAT TREATMENTS.

| Specimen | Structure | Approximate Transformation, per cent | Heat Treatment after Austenitizing[a] | Rockwell Hardness, C Scale |
|---|---|---|---|---|
| 40 | Proeutectoid ferrite formed at 1300 F | 10[b] | 4 min at 1300 F | 58.5 |
| 41 | Proeutectoid ferrite and pearlite | 30 | 35 min at 1300 F | 50.3 |
| 42 | Proeutectoid ferrite and pearlite formed at 1300 F | 100 | 3 hr at 1300 F | 9.3 |
| 57 | Proeutectoid ferrite and pearlite formed at 1200 F | 40 | 2 min at 1200 F | 46.1 |
| 58 | Proeutectoid ferrite and pearlite formed at 1200 F | 100 | 15 min at 1200 F | 27.2 |
| 43 | Proeutectoid ferrite and trace of pearlite formed at 1100 F | 10 | 4 min at 1100 F | 57.6 |
| 44 | Proeutectoid ferrite and pearlite formed at 1100 F | 30 | 9 min at 1100 F | 51.8 |
| 45 | Proeutectoid ferrite and pearlite formed at 1100 F | 100 | 30 min at 1100 F | 31.9 |
| 46 | Bainite formed at 900 F | 20 | 30 sec at 900 F | 52.3 |
| 47 | Bainite—pearlite[c] | 95 | 5 hr at 900 F | 28.9 |
| 48 | Bainite formed at 700 F | 30 | 50 sec at 700 F | 54.8 |
| 49 | Bainite formed at 700 F | 100 | 15 min at 700 F | 40.6 |
| 50 | Martensite | ... | Brine-quenched | 59.9 |
| 51 | Martensite tempered at 1300 F | ... | Brine-quenched, 1 hr. at 1300 F | 26.5 |
| 52 | Martensite tempered at 1100 F | ... | Brine-quenched, 1 hr at 1100 F | 34.6 |
| 53 | Martensite tempered at 900 F | ... | Brine-quenched, 1 hr at 900 F | 40.7 |
| 54 | Martensite tempered at 700 F | ... | Brine-quenched, 1 hr at 700 F | 45.8 |
| 55 | Martensite tempered at 500 F | ... | Brine-quenched, 1 hr at 500 F | 50.0 |
| 59 | Martensite tempered at 400 F | ... | Brine-quenched, 1 hr at 400 F | 54.5 |
| 60 | Martensite tempered at 300 F | ... | Brine-quenched, 1 hr at 300 F | 58.1 |

[a] Specimens were austenitized 30 min at 1800 F, then quenched from the final temperature indicated.

[b] Numbers refer to approximate percentage transformed. When not 100 per cent transformed, the balance is martensite.

[c] "Degenerate pearlite" formed during a secondary reaction at this temperature.

## EXPERIMENTAL WORK

Specimens $\frac{1}{8}$ in. thick were cut from 1 by 1 in. bar stock of an AISI 4140 steel. The composition is as follows:

| | |
|---|---|
| Carbon, per cent | 0.40 |
| Manganese, per cent | 0.77 |
| Silicon, per cent | 0.29 |
| Phosphorus, per cent | 0.015 |
| Sulfur, per cent | 0.030 |
| Chromium, per cent | 1.01 |
| Molybdenum, per cent | 0.12 |

Two mutually perpendicular V-notches were machined on these $\frac{1}{8}$ by 1 by 1 in. pieces so that they would be broken easily after heat treatment into quarters for distribution to the subcommittee. Each member examined only the center of the quarter section to avoid any possible segregation of inclusions at the surface or center of the bar.

The notched $\frac{1}{8}$ by 1 by 1 in. specimens were copper-plated and then austenitized 30 min at 1800 F in air in a tube furnace at the U. S. Steel Fundamental Research Laboratory. A duplex grain size, ASTM

No. 4 and No. 7, was obtained by this treatment. Isothermal transformations at temperatures ranging from 700 to 1300 F were performed by transferring specimens from the austenitizing furnace to lead pots at the desired temperatures, holding for times required to obtain partial or complete transformation, and quenching in brine. Other specimens were quenched in brine from the austenitizing furnace to form martensite and then tempered in the lead pots. Heat treatments used to produce the various structures are listed in Table I with the corresponding hardness values.

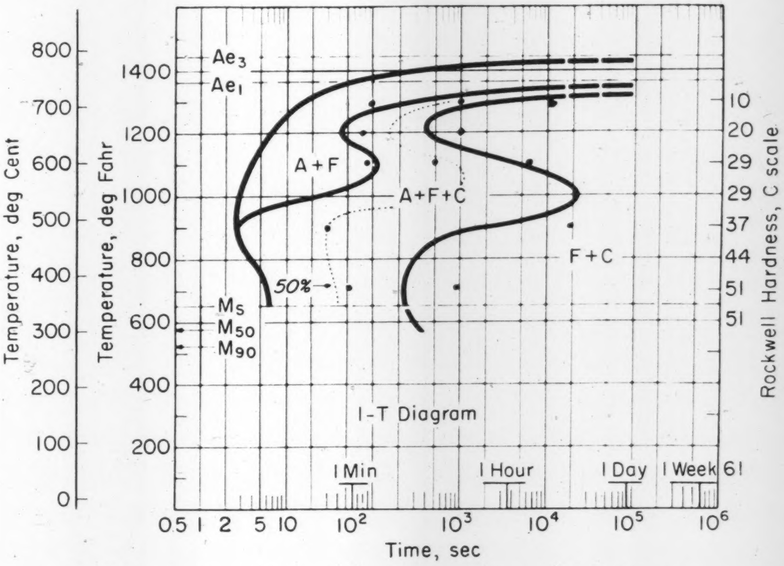

Fig. 1.—Isothermal Transformation Diagram of AISI 4140 Steel.

For light and electron metallographic examination, the specimens were polished mechanically, using alternate polishing and etching during finishing to eliminate disturbed metal at the surface. Care was taken to avoid heating of the specimens during mounting, polishing, and etching. The specimens were etched for a few seconds up to ½ min in Super-picral[2] etching solution. It is not possible to prescribe an exact etching time for electron metallographic work because proper etch depth is very critical, and small variations in temperature, concentration, and age of etchants can result in wide variations in the time required to achieve an optimum etch. Optical photomicrographs were taken of each structure at the U. S. Steel Fundamental Research Laboratory at a magnification of 2500 diameters.

Following the technique described in previous reports (**1**, **2**, **3**, **4**), most of the electron microscope observations were made on metal shadowed-negative plastic replicas. The illustrations are from positive prints made by direct enlargement from the plates to the magnifications shown.

---

[2] Formula:
100 ml saturated solution of picric acid in 95 per cent methyl alcohol
2 ml of 12.8 per cent aqueous solution of Zephiran chloride

Figure 1 shows a time-temperature-transformation diagram for a heat of AISI 4140 steel quite similar in composition to the steel used. The marked points indicate the aim of the various heat treatments.

Because of the similarity in the nature and organization of this work with that done on the series of specimens of eutectoid steel, this presentation follows closely that adopted for the First Progress Report, Subcommittee XI (1). That is, the isothermal transformation structures will be discussed in order of decreasing transformation temperature, starting with coarse pearlite, and the tempered structures will be discussed in order of increasing tempering temperature.

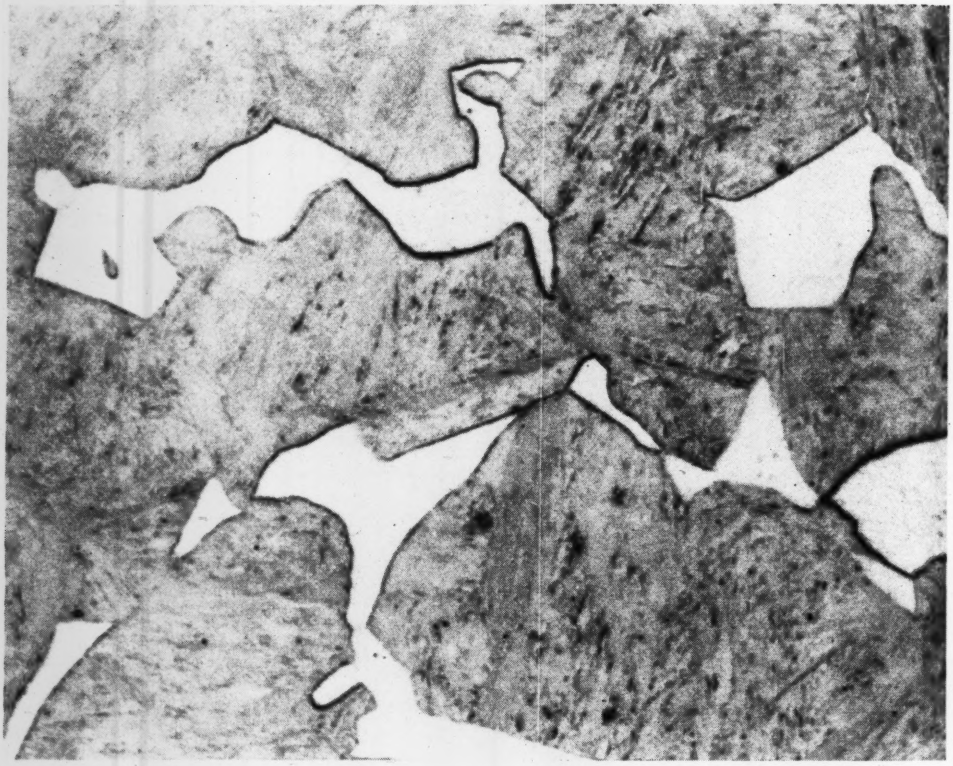

FIG. 2.—Light Micrograph of Proeutectoid Ferrite and Martensite Resulting from Partial Transformation at 1300 F (×2500).

## PEARLITE

During the previous investigation of eutectoid carbon steel, the subcommittee found that examination of specimens partially transformed to bainite provides considerable insight into the nature of the structure when compared to fully transformed structures. In this investigation of 4140 steel, the same approach has been extended to the pearlite region, and it is particularly helpful here because of the proeutectoid ferrite that forms in this steel.

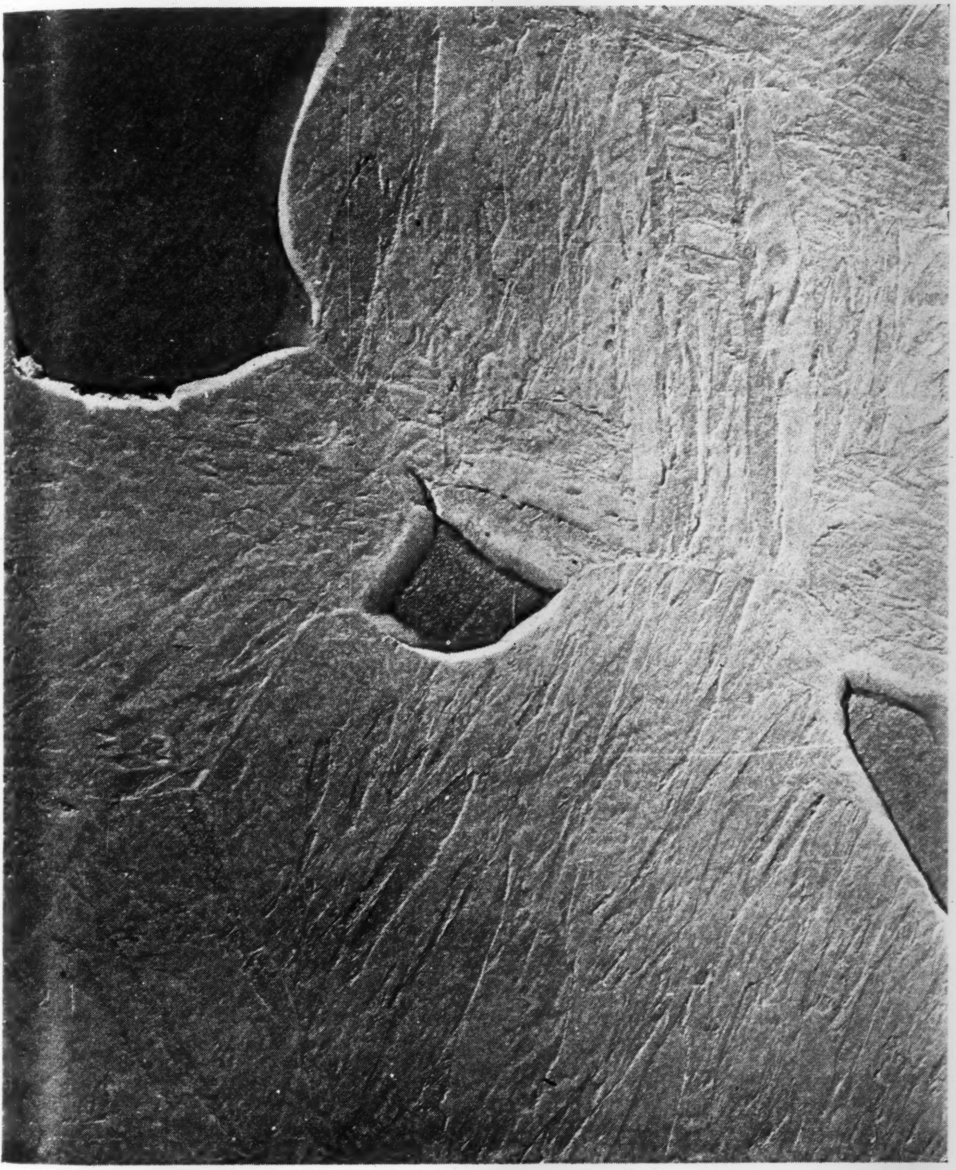

FIG. 3.—Electron Micrograph of Proeutectoid Ferrite and Martensite Resulting from Partial Transformation at 1300 F (×15,000).

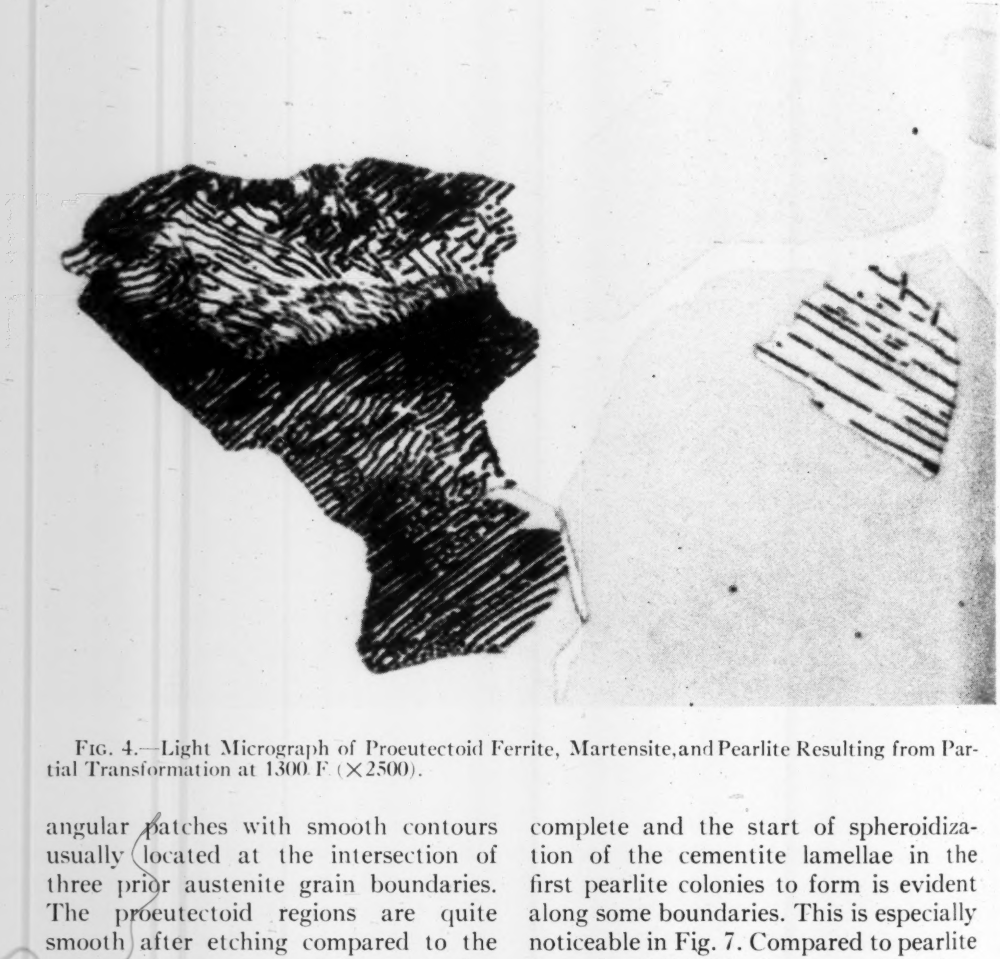

FIG. 4.—Light Micrograph of Proeutectoid Ferrite, Martensite, and Pearlite Resulting from Partial Transformation at 1300 F (×2500).

*Isothermal Transformation at 1300 F:*

The first stage of austenite decomposition at 1300 F is illustrated in the light and electron micrographs of Figs. 2 and 3 showing specimen 40, which was held 4 min at that temperature. Sections through the proeutectoid ferrite are angular patches with smooth contours usually located at the intersection of three prior austenite grain boundaries. The proeutectoid regions are quite smooth after etching compared to the roughened appearance of the martensite.

Figures 4 and 5 show light and electron micrographs of specimen 41 held 35 min at 1300 F. These micrographs reveal that this holding time was sufficient to form some pearlite colonies. The pearlite is coarse and the growing edges of the lamellae appear to extend to the boundaries (martensite). The proeutectoid ferrite has the same appearance as it had in specimen 40.

Specimen 42 was held 3 hr at 1300 F and, as can be seen in Figs. 6 and 7, the formation and growth of pearlite is complete and the start of spheroidization of the cementite lamellae in the first pearlite colonies to form is evident along some boundaries. This is especially noticeable in Fig. 7. Compared to pearlite formed at 1300 F in the eutectoid carbon steel, the pearlite in AISI 4140 steel is much finer but has a wide range of carbide spacings. Many of the cementite lamellae are quite discontinuous and there are frequently several carbide orientations in a single colony.

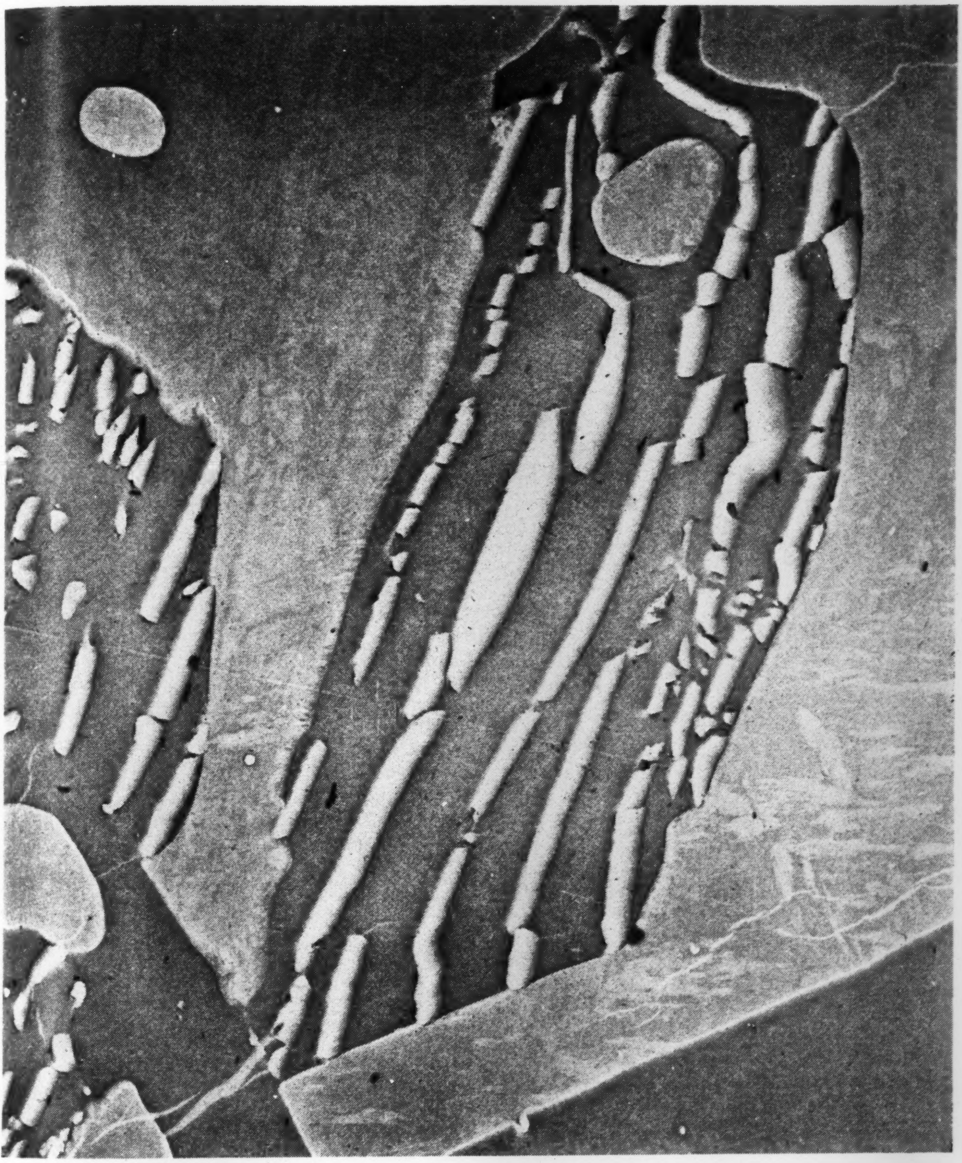

FIG. 5.—Electron Micrograph of Proeutectoid Ferrite, Martensite, and Pearlite Resulting from Partial Transformation at 1300 F (×15,000).

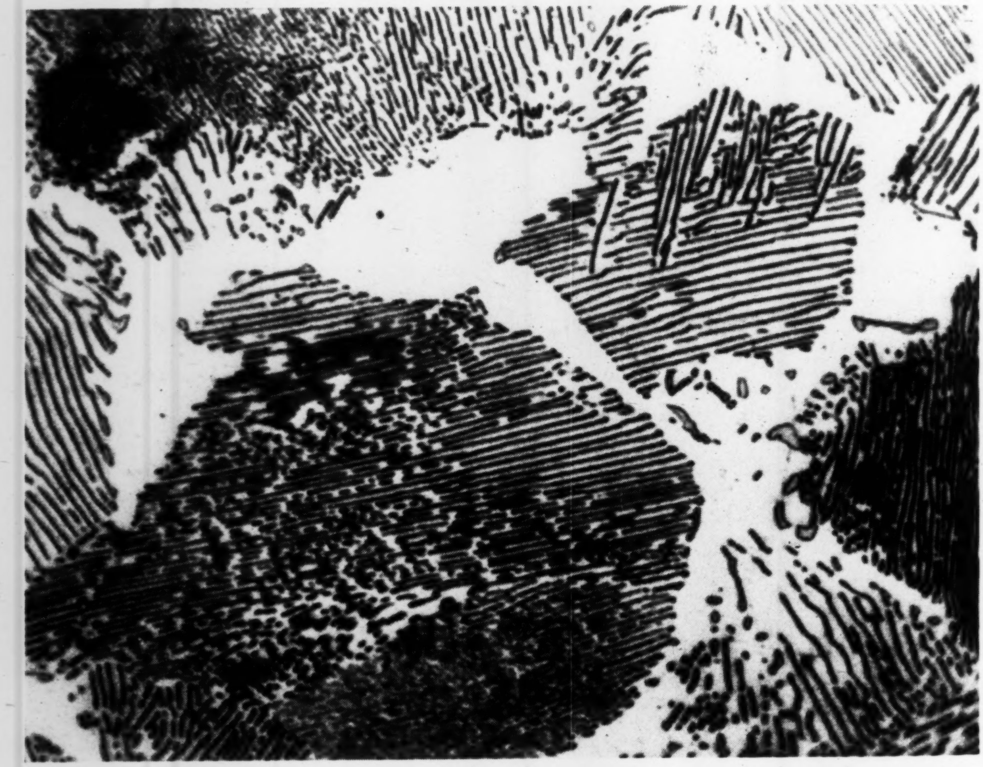

FIG. 6.—Light Micrograph of Coarse Pearlite Formed at 1300 F (×2500).

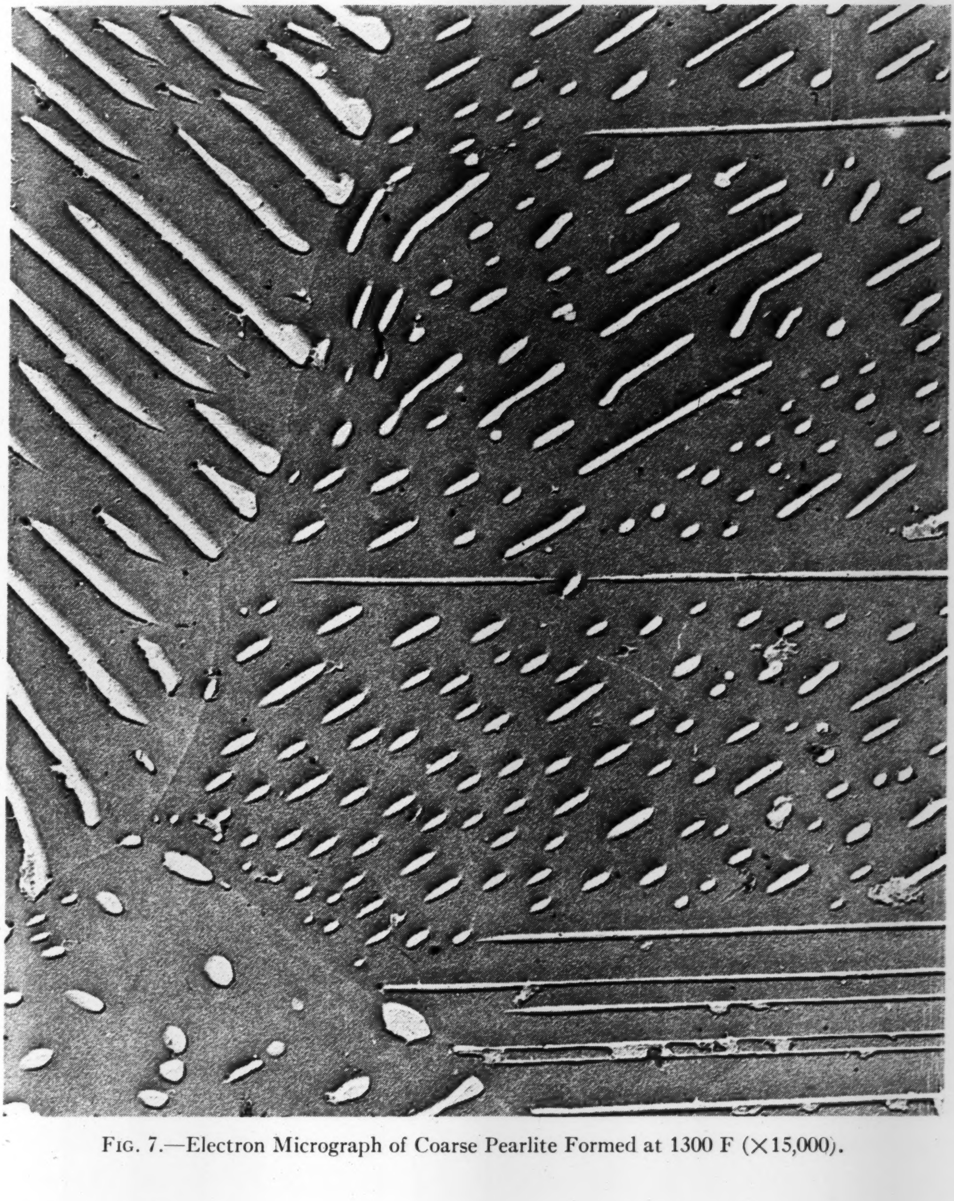

FIG. 7.—Electron Micrograph of Coarse Pearlite Formed at 1300 F (×15,000).

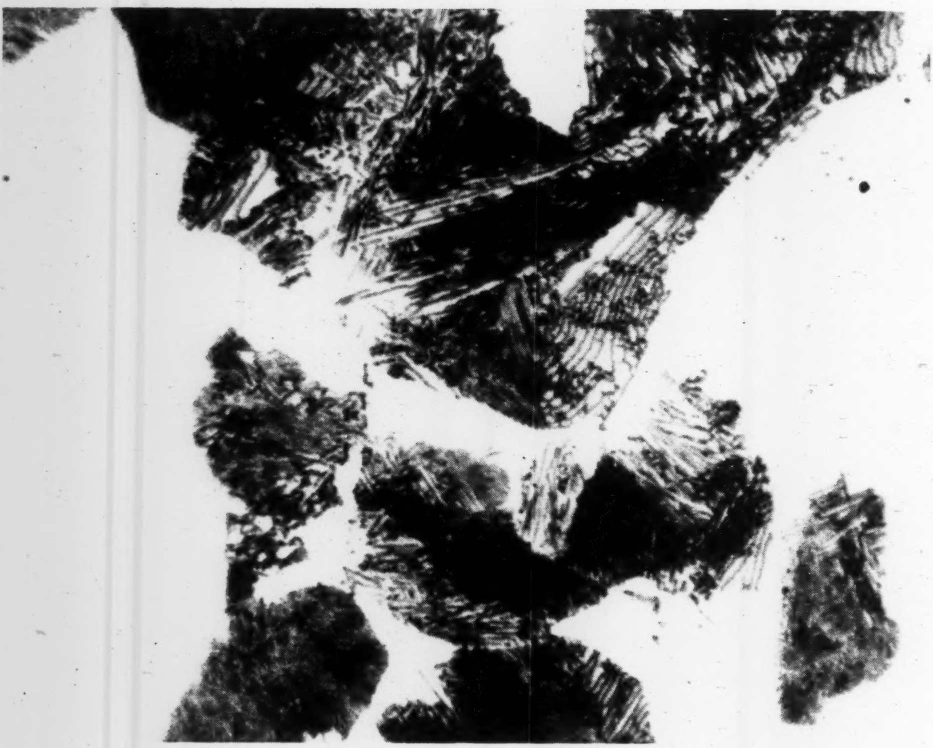

FIG. 8.—Light Micrograph of Pearlite and Martensite Resulting from Partial Transformation at 1200 F (×2500).

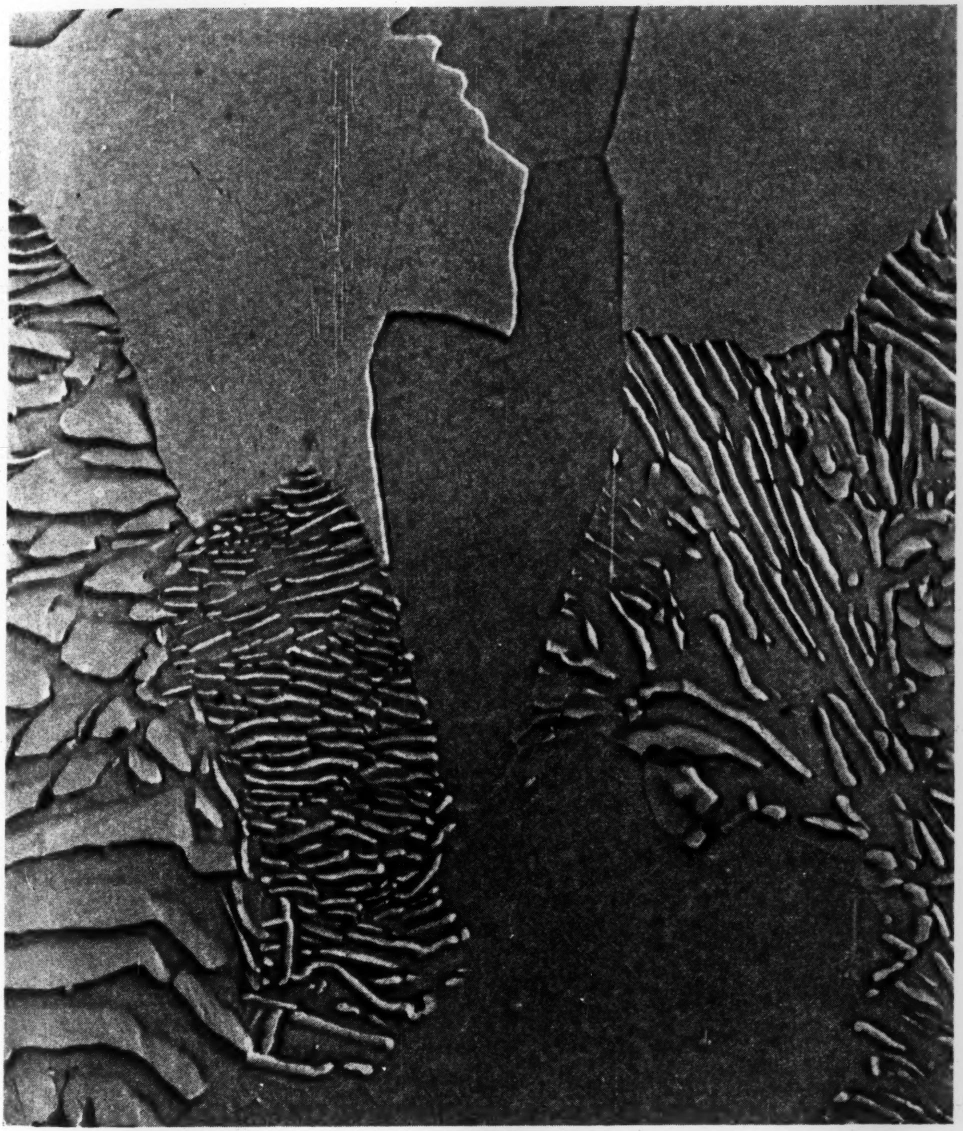

FIG. 9.—Electron Micrograph of Pearlite and Martensite Resulting from Partial Transformation at 1200 F (×15,000).

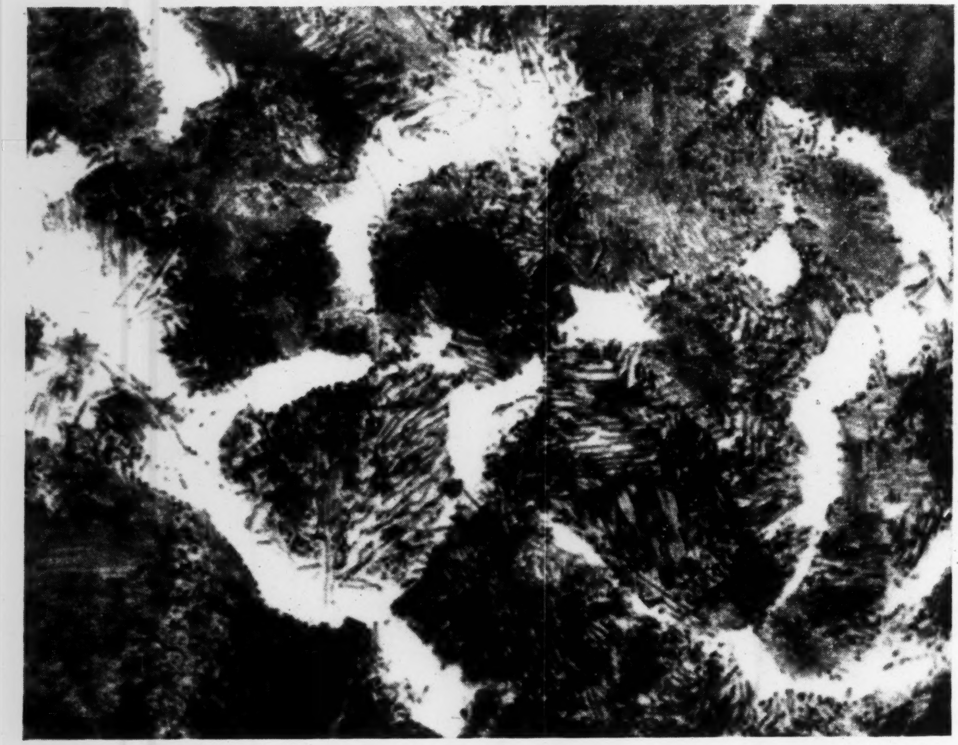

FIG. 10.—Light Micrograph of Pearlite Formed at 1200 F (×2500).

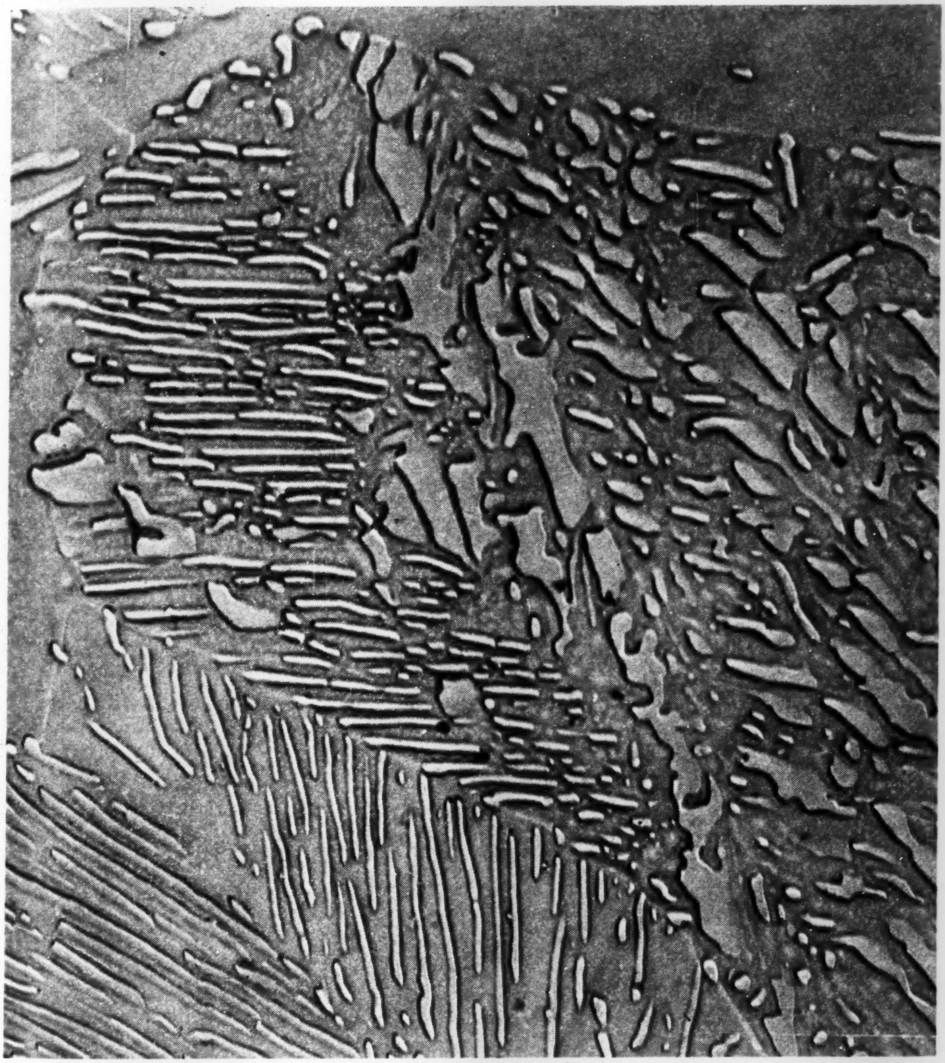

FIG. 11.—Electron Micrograph of Pearlite Formed at 1200 F (×15,000).

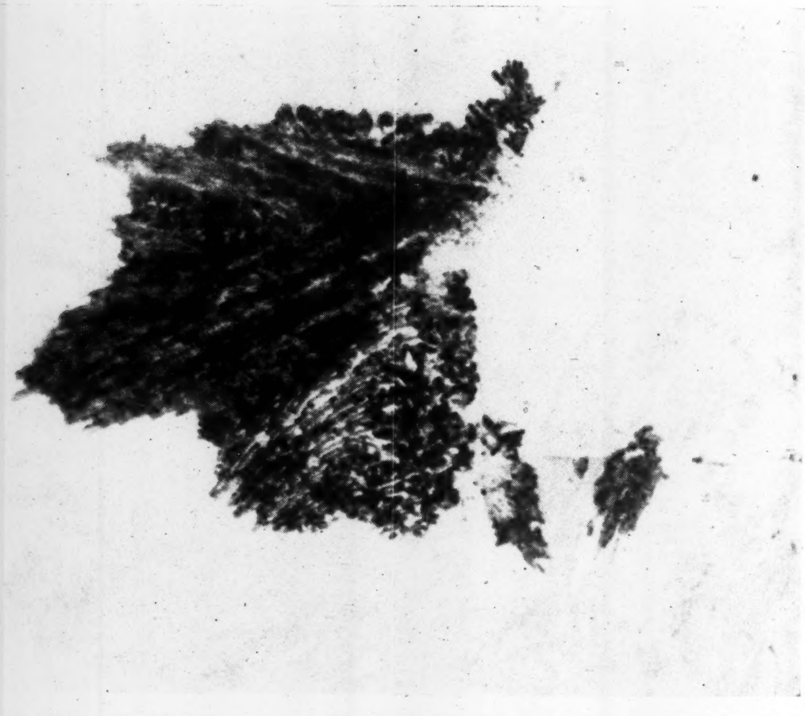

FIG. 12.—Light Micrograph of Proeutectoid Ferrite, Pearlite, and Martensite Resulting from Partial Transformation at 1100 F (×2500).

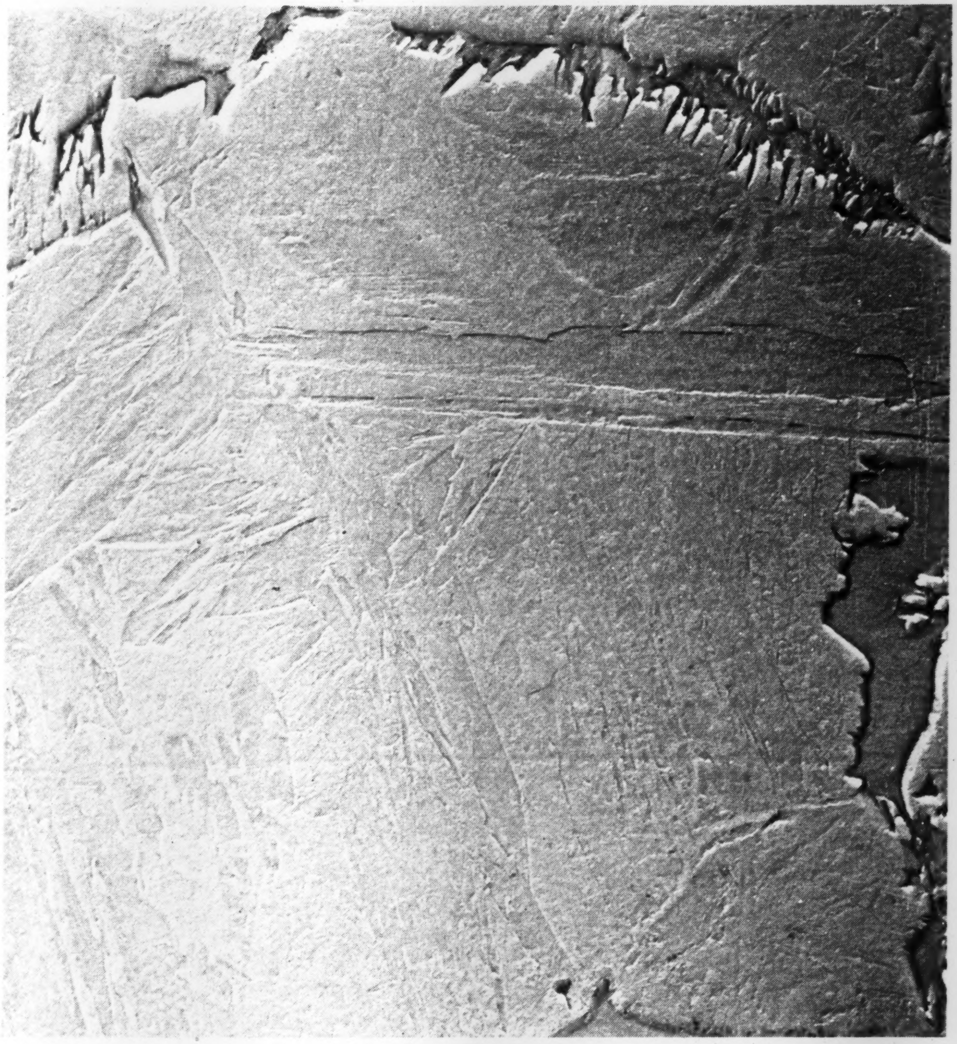

FIG. 13.—Electron Micrograph of Proeutectoid Ferrite, Pearlite, and Martensite Resulting from Partial Transformation at 1100 F (×7500).

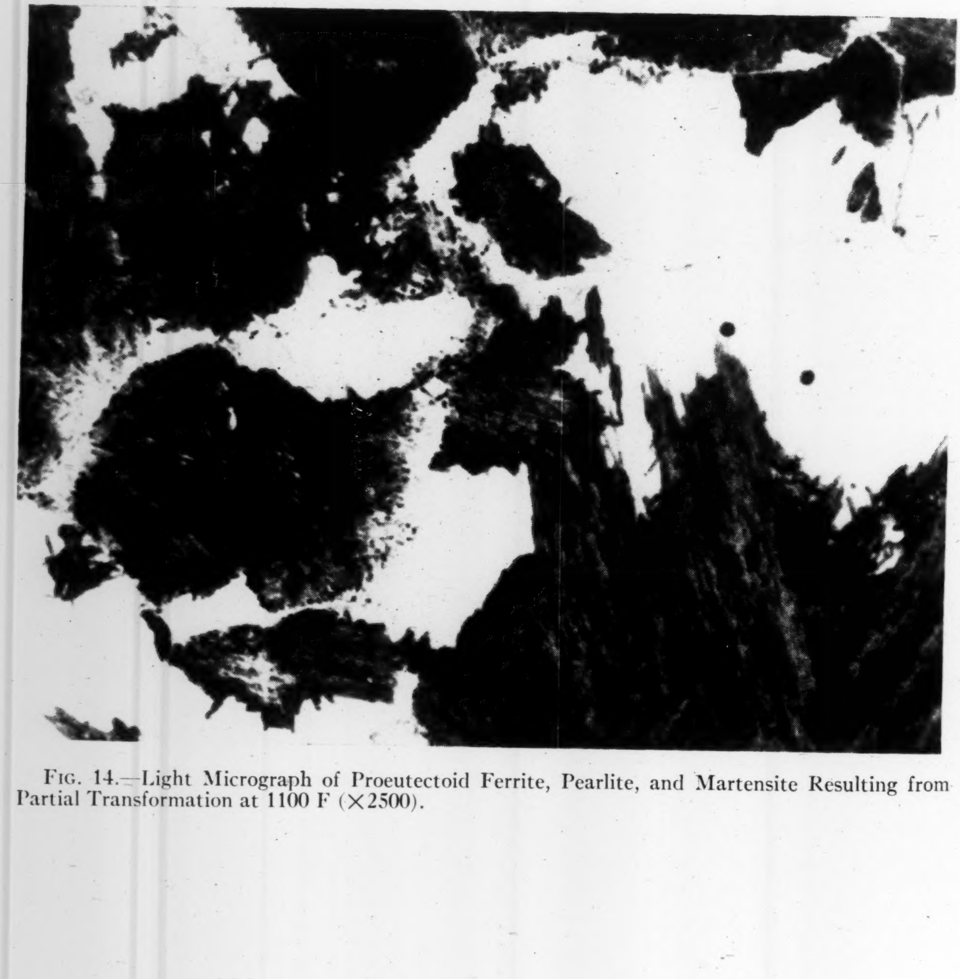

FIG. 14.—Light Micrograph of Proeutectoid Ferrite, Pearlite, and Martensite Resulting from Partial Transformation at 1100 F (×2500).

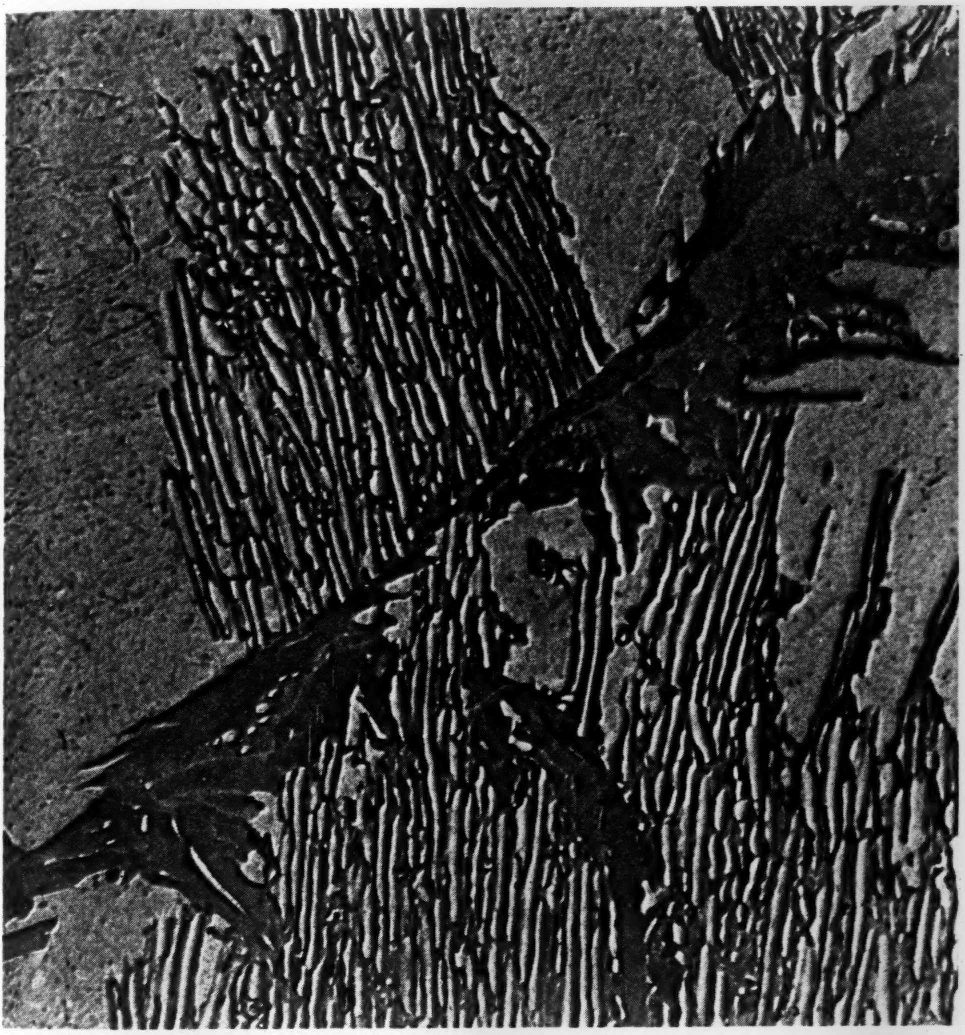

FIG. 15.—Electron Micrograph of Proeutectoid Ferrite, Pearlite, and Martensite Resulting from Partial Transformation at 1100 F (×15,000).

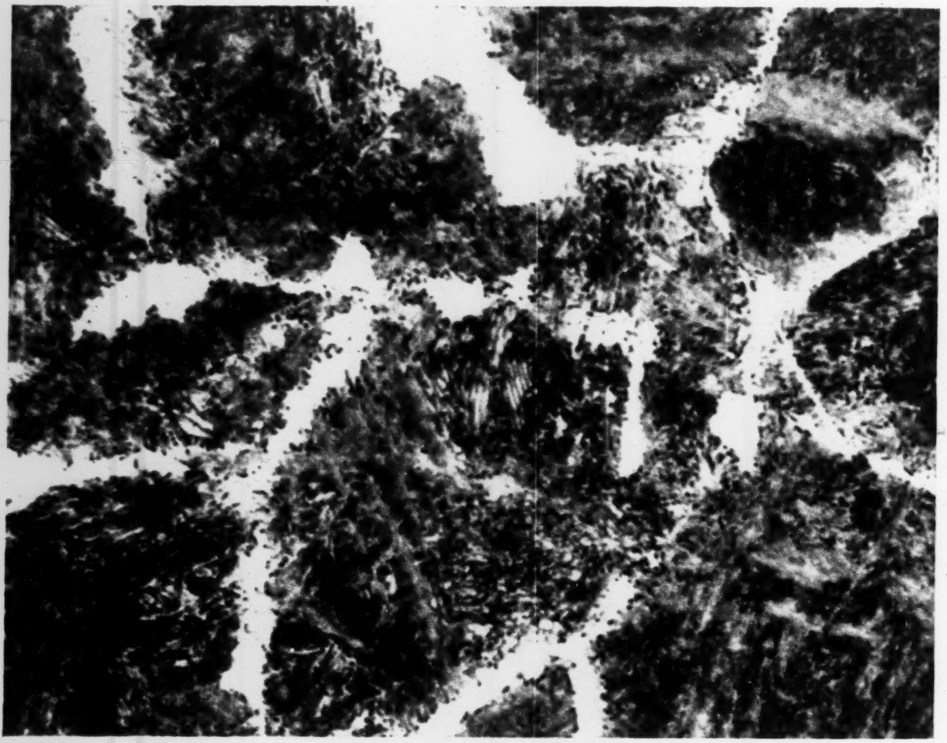

FIG. 16.—Light Micrograph of Pearlite Formed at 1100 F (×2500).

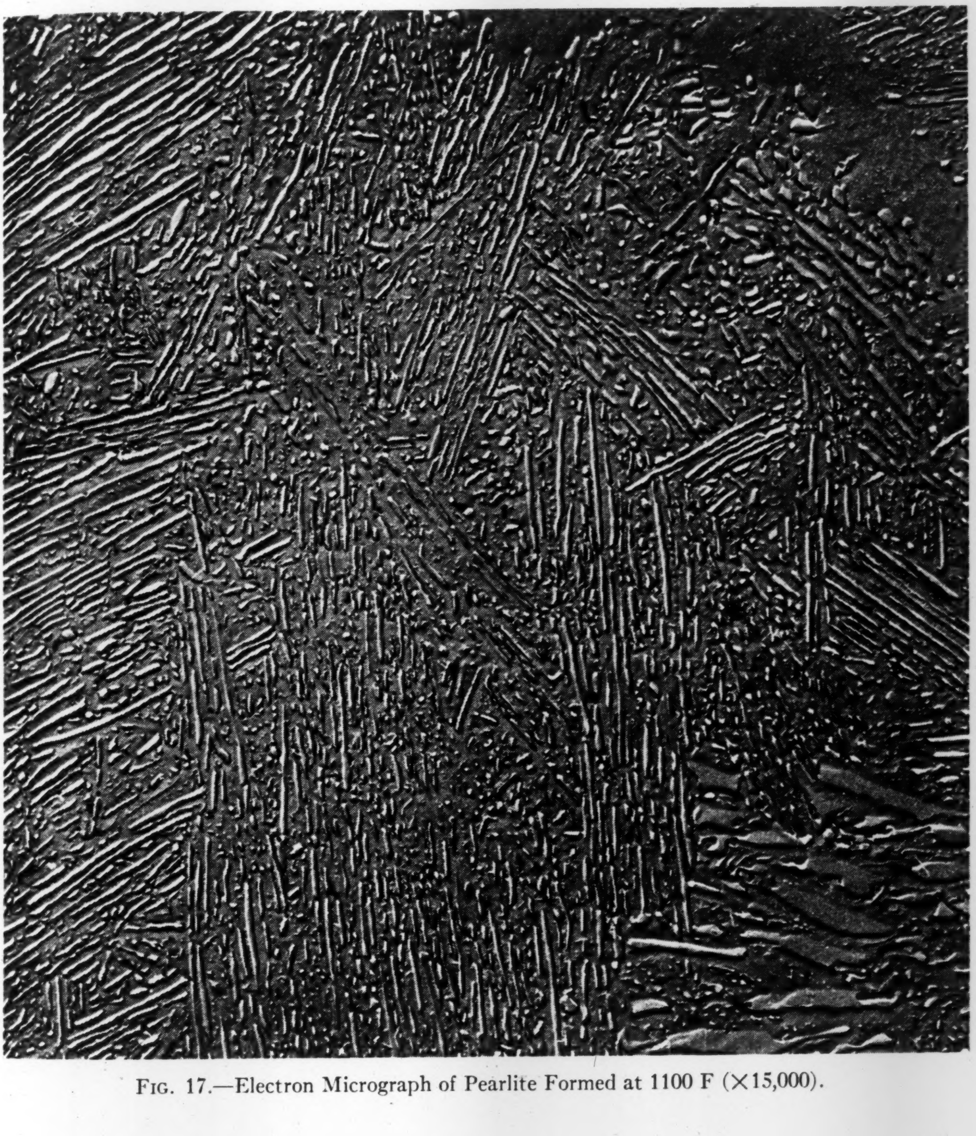

FIG. 17.—Electron Micrograph of Pearlite Formed at 1100 F (×15,000).

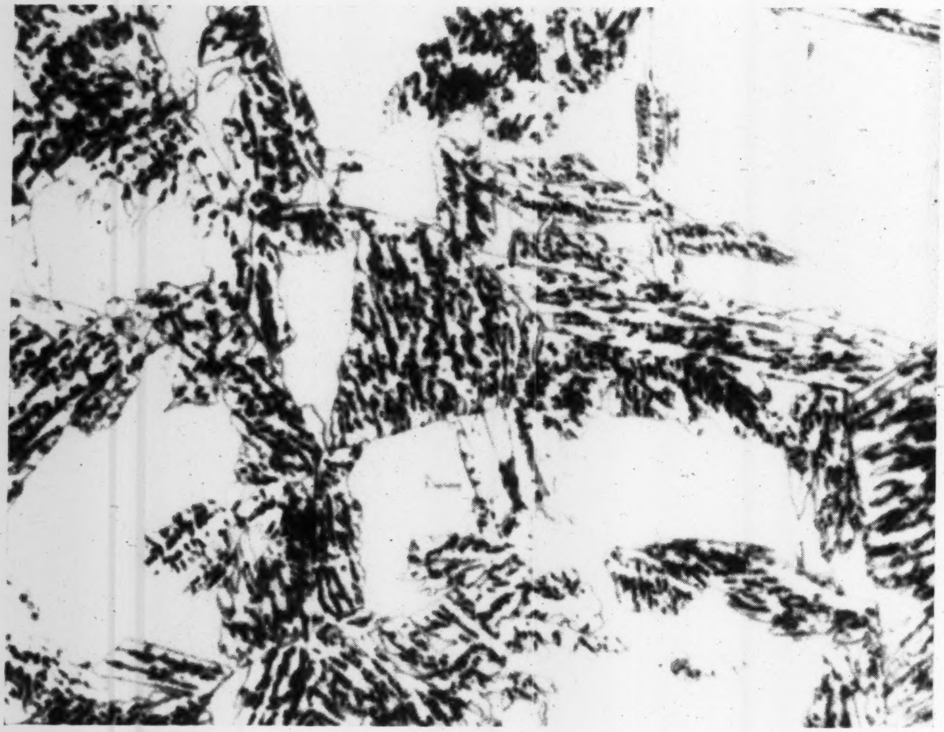

FIG. 18.—Light Micrograph of Bainite and Martensite Resulting from Partial Transformation at 900 F (×2500).

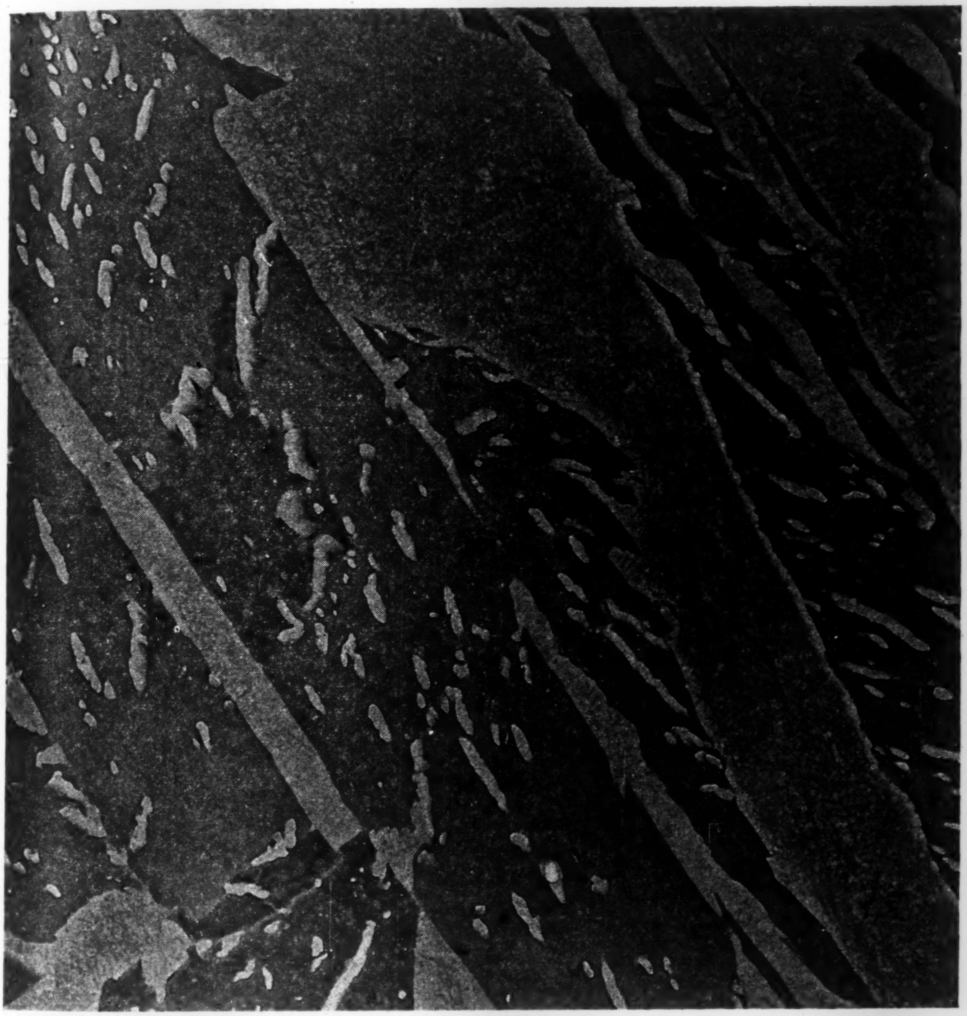

FIG. 19.—Electron Micrograph of Bainite and Martensite Resulting from Partial Transformation at 900 F (×15,000).

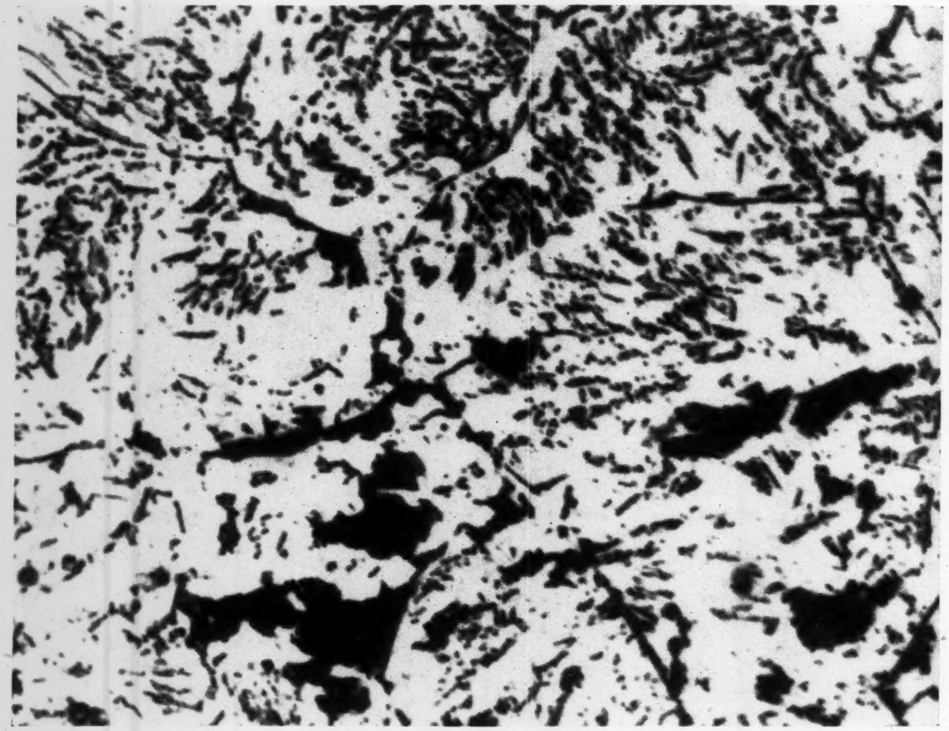

FIG. 20.—Light Micrograph of Bainite and Pearlite Resulting from Transformation at 900 F (×2500).

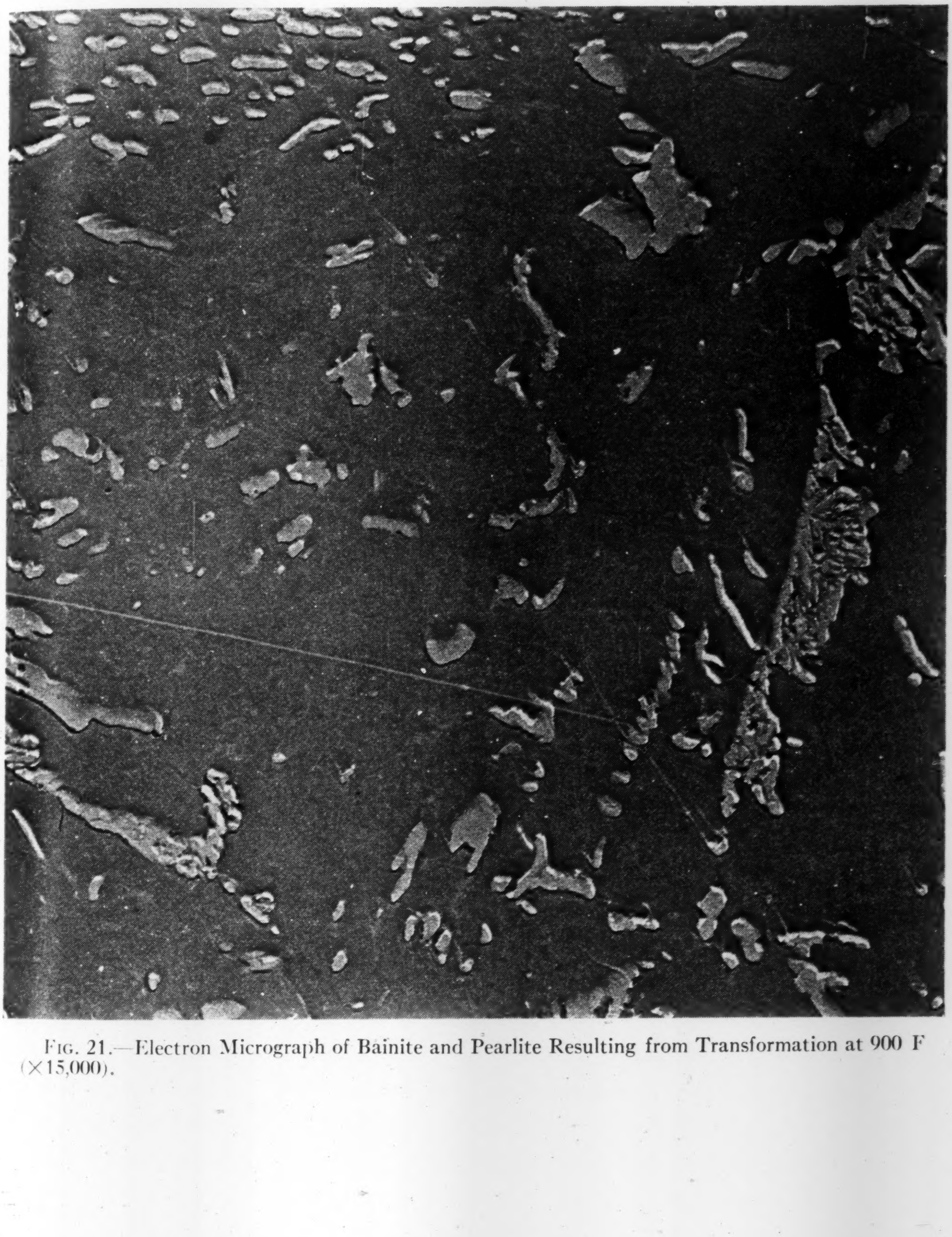

FIG. 21.—Electron Micrograph of Bainite and Pearlite Resulting from Transformation at 900 F (×15,000).

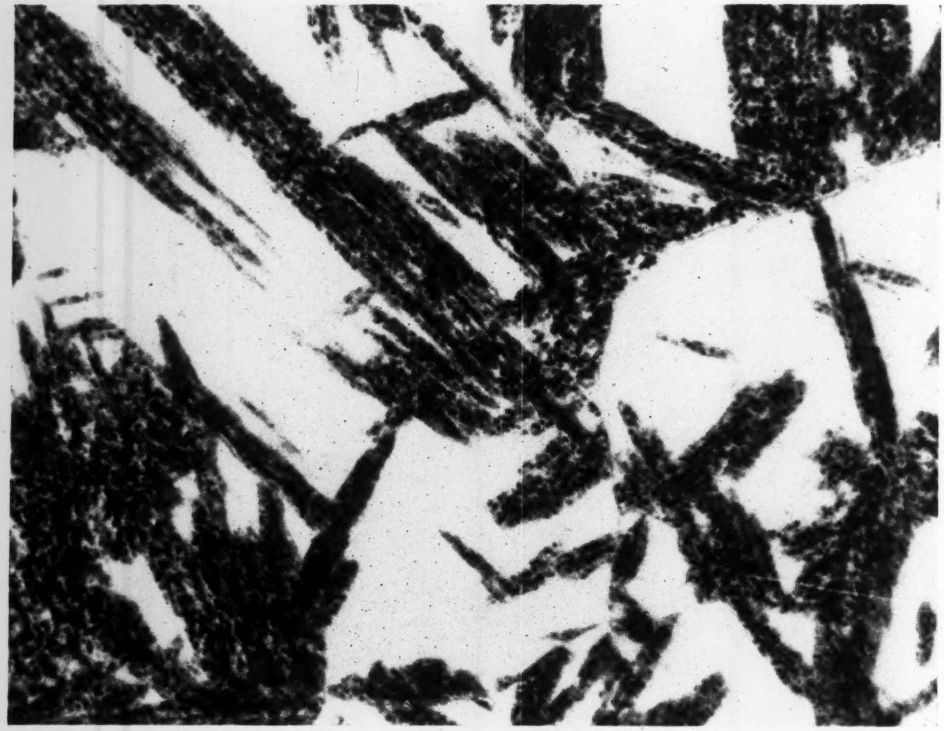

FIG. 22.—Light Micrograph of Bainite and Martensite Resulting from Partial Transformation at 700 F (×2500).

### *Isothermal Transformation at 1200 F:*

Specimen 57 was heated only 2 min at 1200 F, but the light and electron micrographs of Figs. 8 and 9 show that the decomposition of austenite is well advanced. Considerable pearlite has formed which is somewhat finer than that formed at 1300 F. There is a slight tendency for the interface between proeutectoid ferrite and martensite to be irregular. The growing edge of pearlite colonies was frequently observed to be serrated with carbide lamellae, surrounded by a sheath of ferrite protruding into the martensite matrix formed on quenching from the transformation temperature. Figures 10 and 11 show pearlite of intermediate fineness produced in specimen 58 by holding 15 min at 1200 F for complete isothermal transformation. Spheroidization of carbide lamellae at grain boundaries is evident in this specimen, similar to the effect noticed in the 1300 F structure.

### *Isothermal Transformation at 1100 F:*

The first stage of austenite decomposition at 1100 F is shown in the light and electron micrographs of Figs. 12 and 13. The structure of specimen 43 held 4 min at 1100 F is seen to consist of a network of proeutectoid ferrite along prior austenite grain boundaries. The interface between ferrite and martensite is extremely jagged and these irregularities develop into pearlitic regions on further holding at this temperature, as shown in Figs. 14 and 15 of specimen 44 heated 9 min at 1100 F. The tendency for the growing edge of the pearlite colony to be serrated, as noted in pearlite forming at 1200 F, is even more marked at 1100 F. Heating for 30 min at 1100 F produces complete transformation, as shown in Figs. 16 and 17 of specimen 45. The pearlite is quite similar to that in the eutectoid steel produced at the same temperature.

## BAINITE

### *Isothermal Transformation at 900 F:*

The structure of specimen 46, which was partially transformed to bainite by holding at 900 F for 30 sec, is shown in the light and electron micrographs of Figs. 18 and 19. The carbide particles in the bainite needles are rather chunky and are much more coarse than in bainite formed in the eutectoid carbon steel. The carbides tend to be oriented in a parallel arrangement across bainite needles somewhat similar to the structure of lower bainite in the eutectoid carbon steel. Narrow bands of martensite appear along the edges of the bainite needles and seem to be very resistant to attack by the etching solution. These zones may be martensite regions of different composition than the remainder, or perhaps may even be films of retained austenite.

Heating for 5 hr is not sufficient to complete transformation at this temperature. Degenerate pearlite is formed as a secondary reaction during heating at 900 F, while carbides formed in bainite needles begin to coalesce along grain boundaries (Figs. 20 and 21).

### *Isothermal Transformation at 700 F:*

Specimen 48, held for 50 sec at 700 F to produce partial transformation above the Ms temperature, is shown in the micrographs of Figs. 22, 23, and 24. Many of the bainite needles appear as long sharp spikes extending out from the prior austenite grain boundaries. The carbide particles are somewhat elongated and tend to be arranged in parallel alignment across the bainite needles. Figures 25 and 26 show the structure of fully transformed bainite produced in specimen 49 by heating 15 min at 700 F. The carbides are considerably coarser than those formed in eutectoid carbon steel at this temperature,

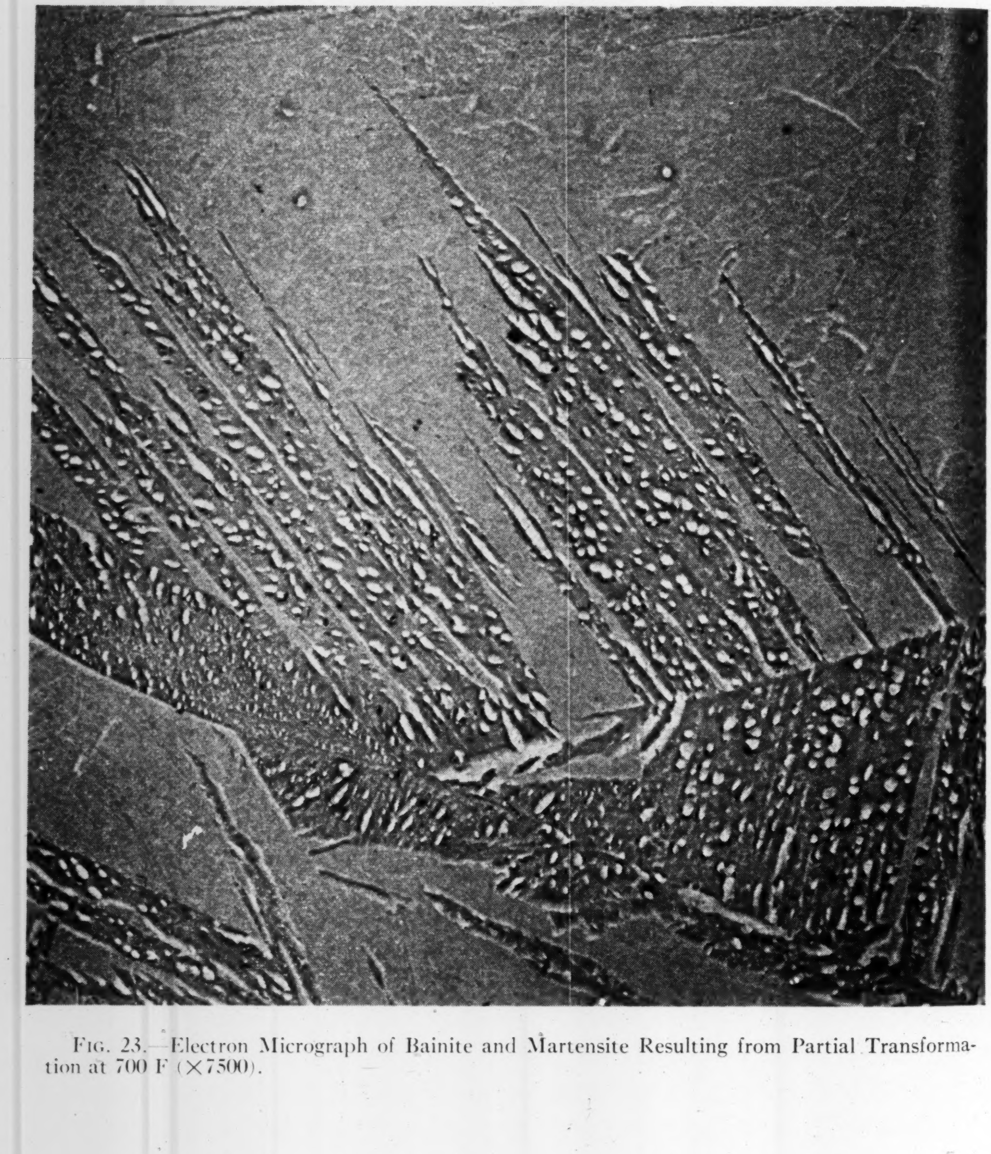

FIG. 23.—Electron Micrograph of Bainite and Martensite Resulting from Partial Transformation at 700 F (×7500).

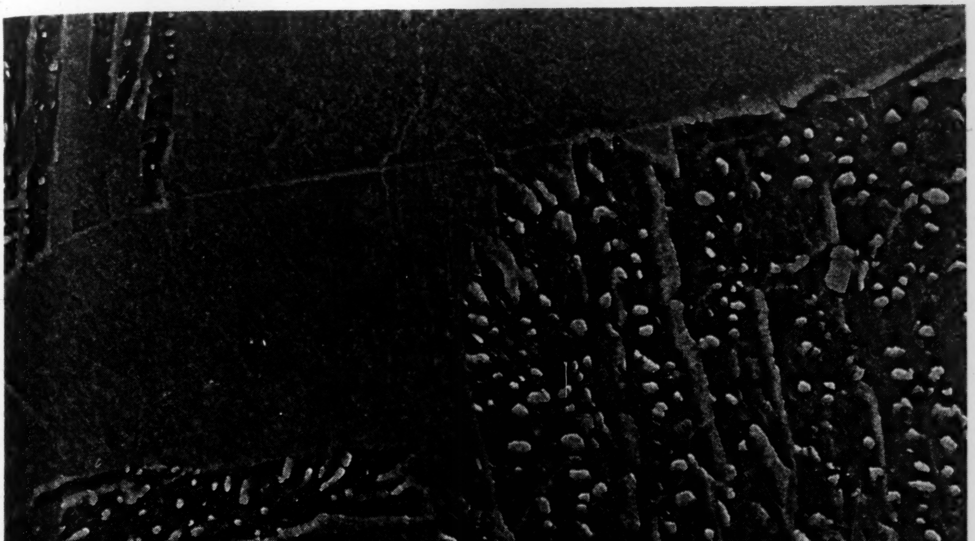

FIG. 24.—Electron Micrograph of Bainite and Martensite Resulting from Partial Transformation at 700 F (×15,000).

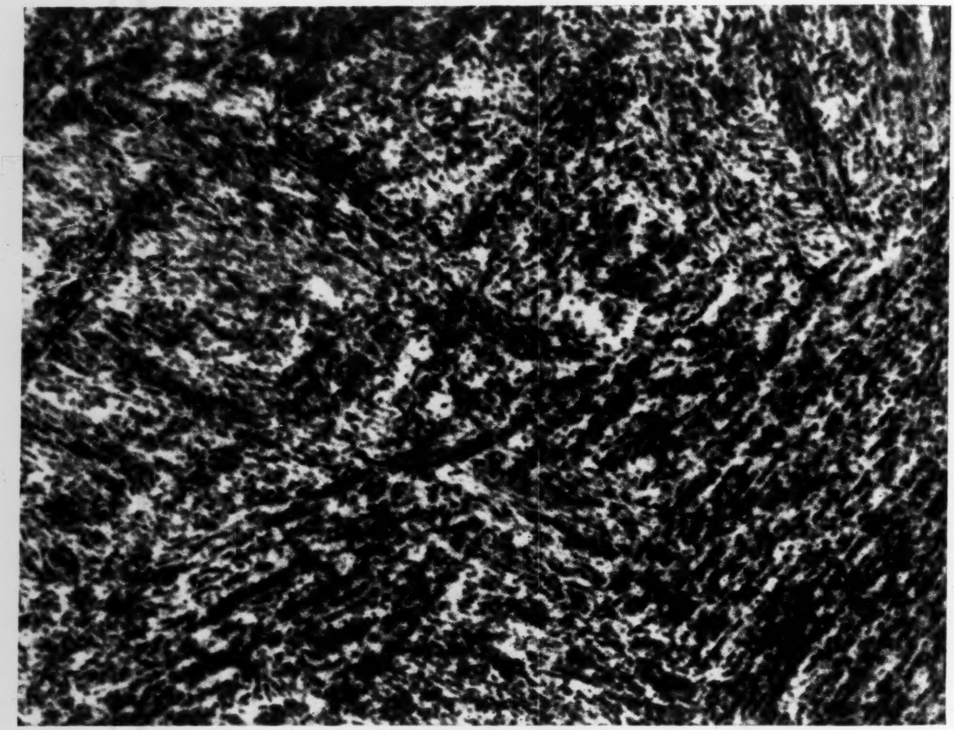

FIG. 25.—Light Micrograph of Bainite Formed at 700 F (×2500).

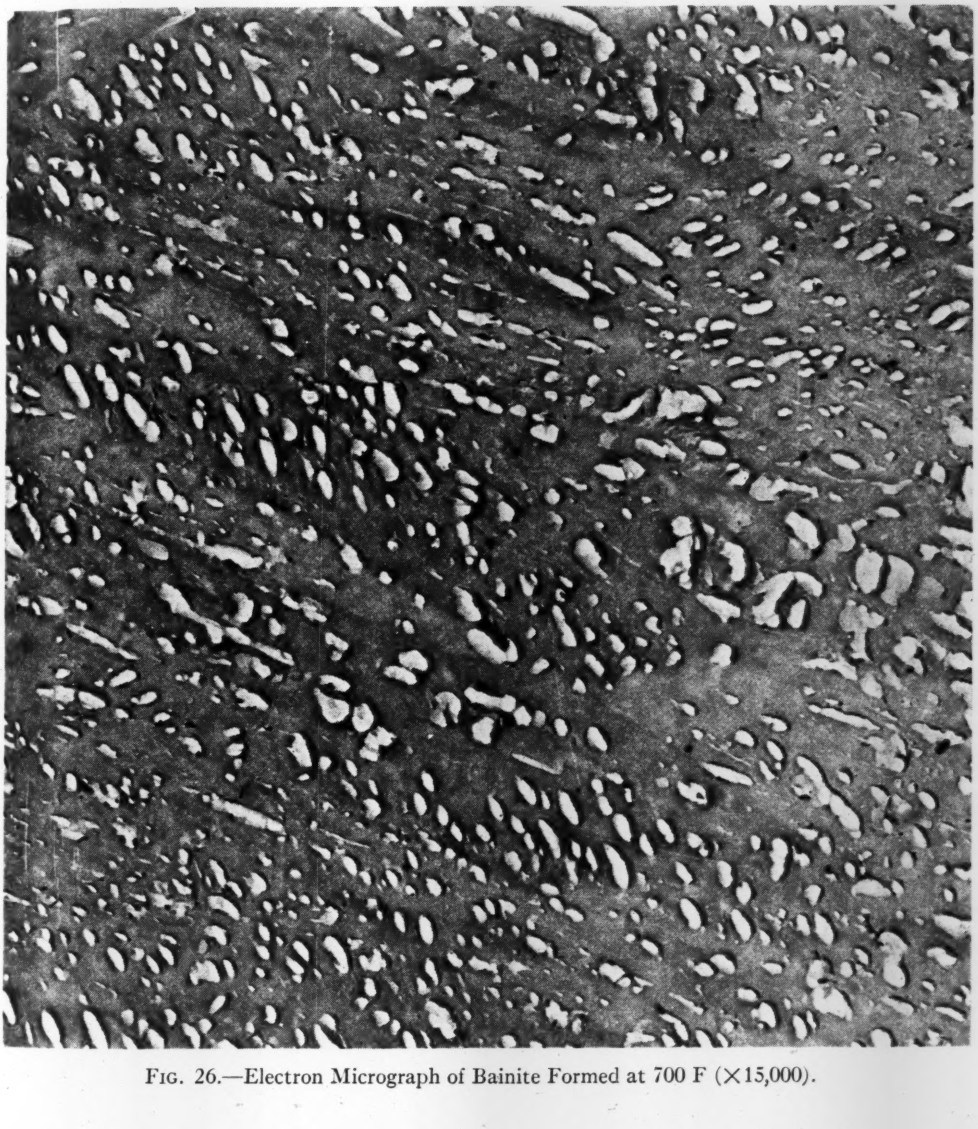

FIG. 26.—Electron Micrograph of Bainite Formed at 700 F (×15,000).

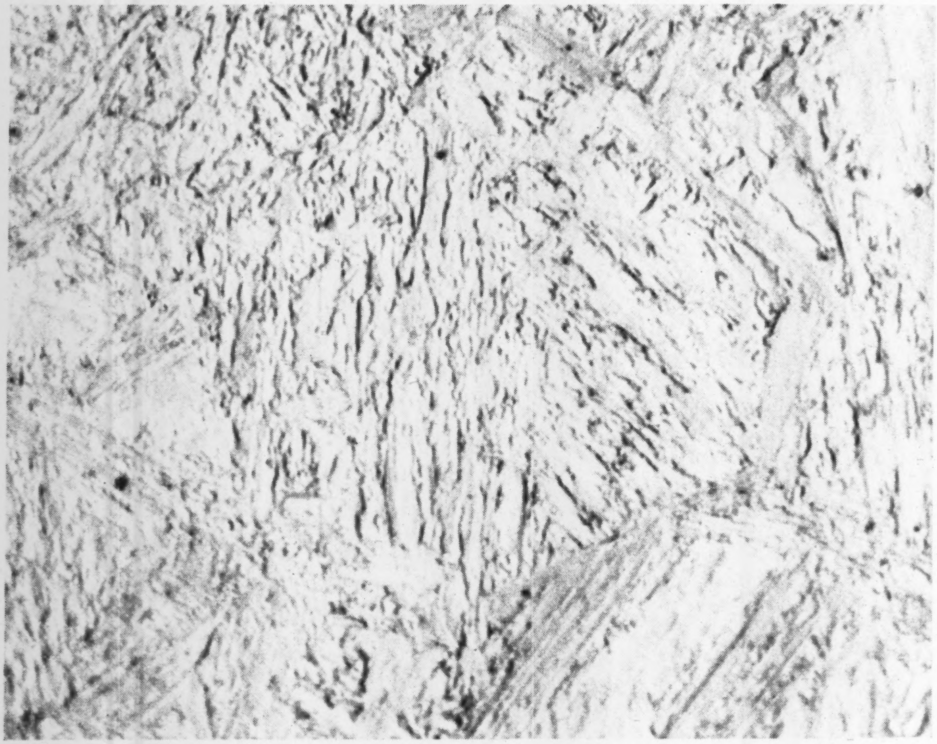

FIG. 27.—Optical Micrograph of Brine-Quenched Martensite (×2500).

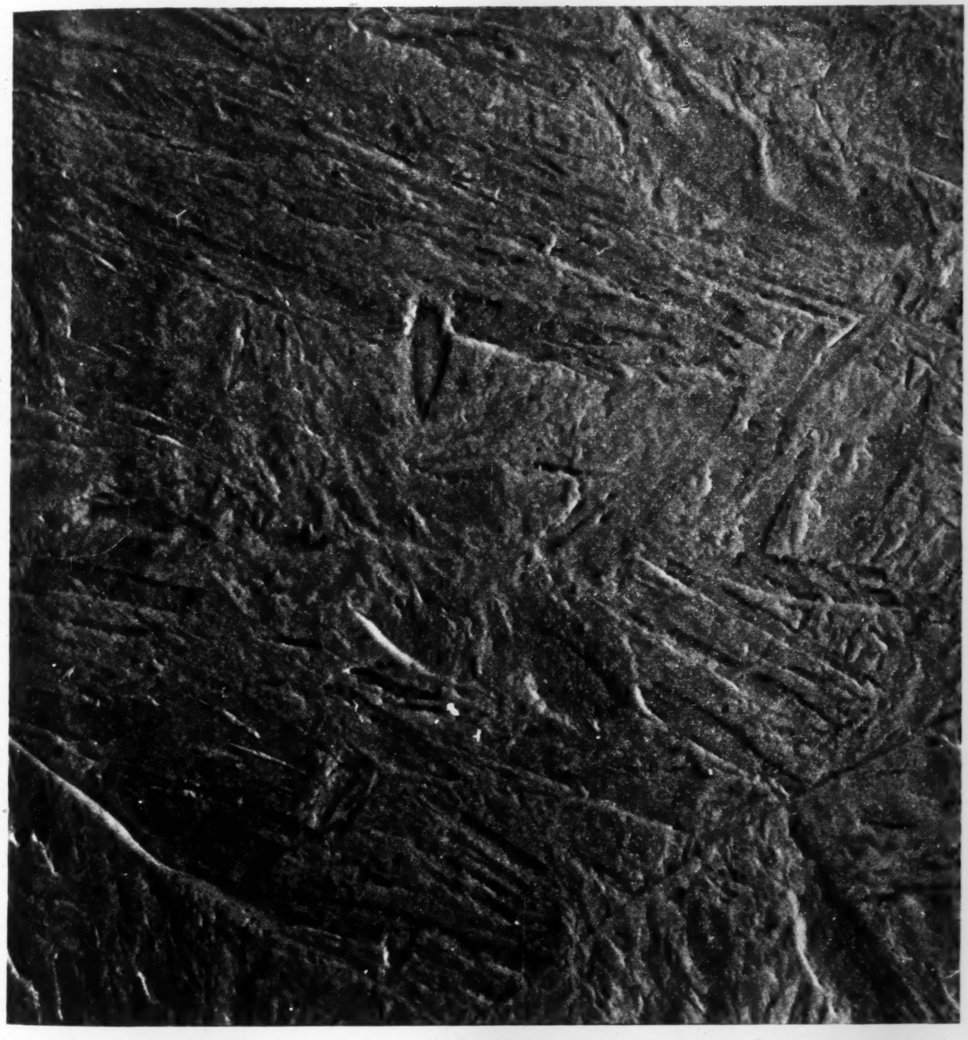

FIG. 28.—Electron Micrograph of Brine-Quenched Martensite (×15,000).

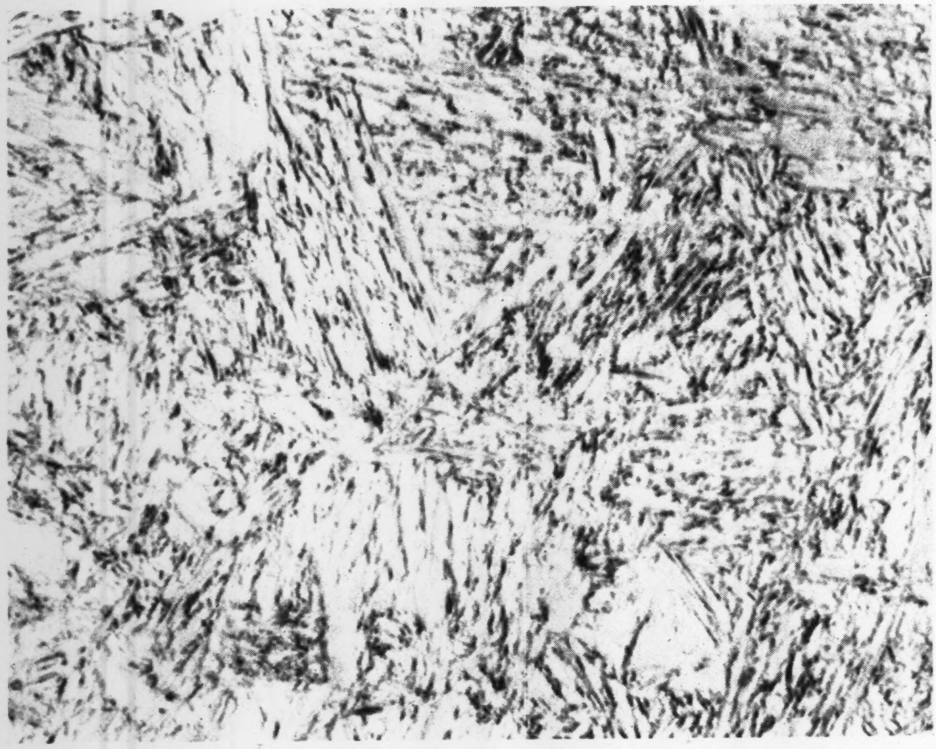

FIG. 29.—Optical Micrograph of Martensite Tempered 1 hr at 300 F (×2500).

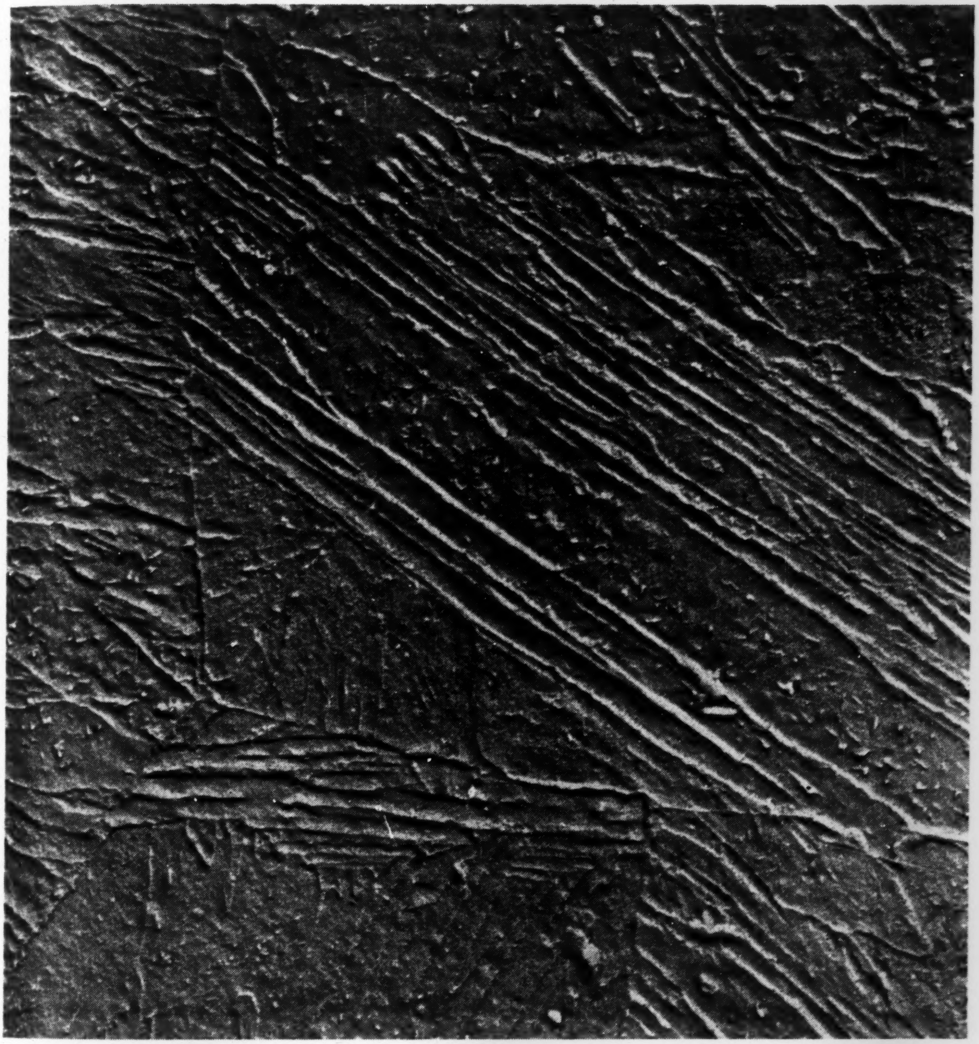

FIG. 30.—Electron Micrograph of Martensite Tempered 1 hr at 300 F (×15,000).

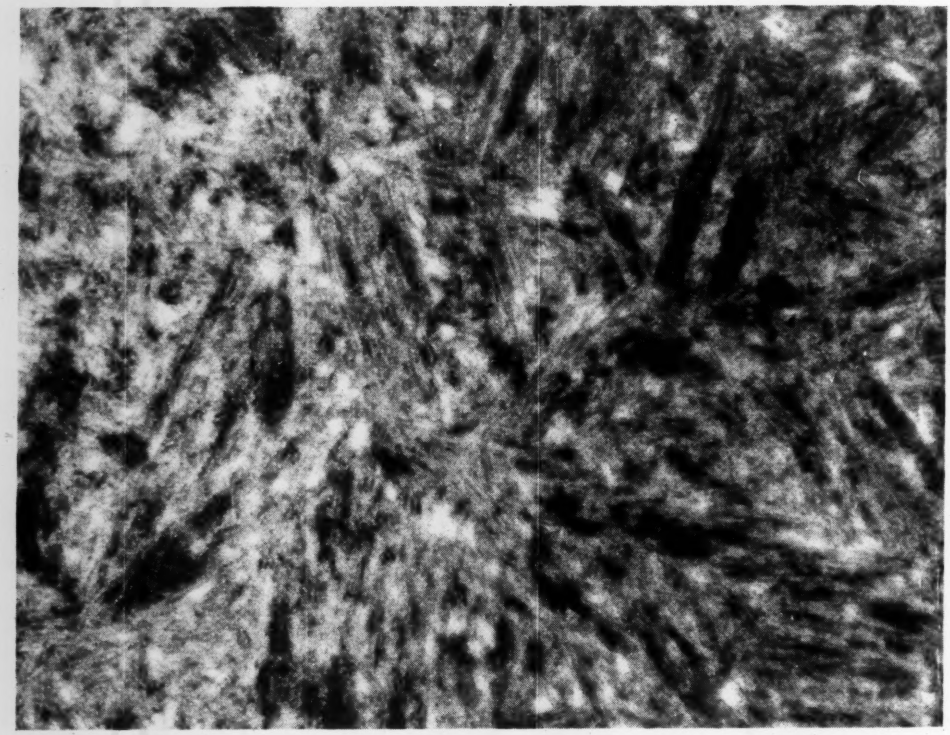

FIG. 31.—Optical Micrograph of Martensite Tempered 1 hr at 400 F (×2500).

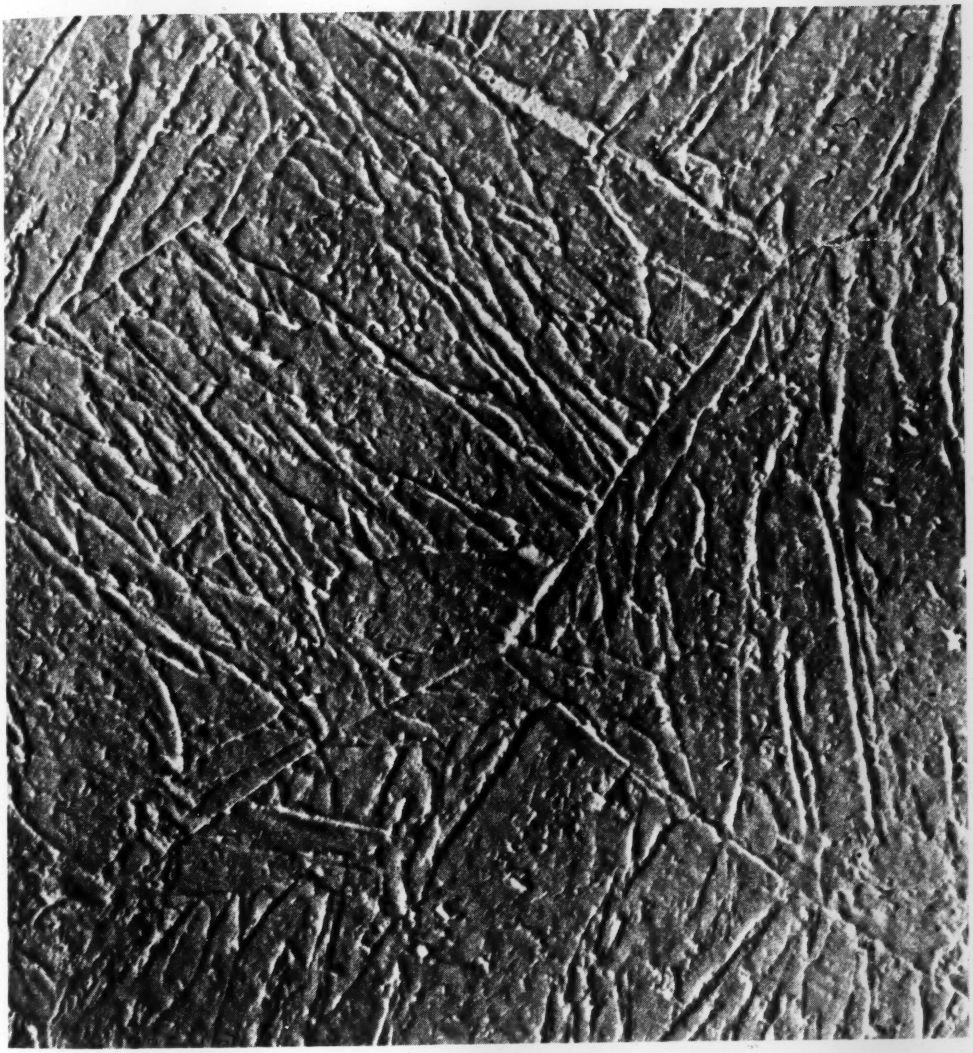

FIG. 32.—Electron Micrograph of Martensite Tempered 1 hr at 400 F (×15,000).

but the arrangement of carbides is somewhat like that in bainite formed at considerably lower temperatures in a 1087 steel. The bainite needle areas are visible because of differential etch attack which is a function of ferrite orientation.

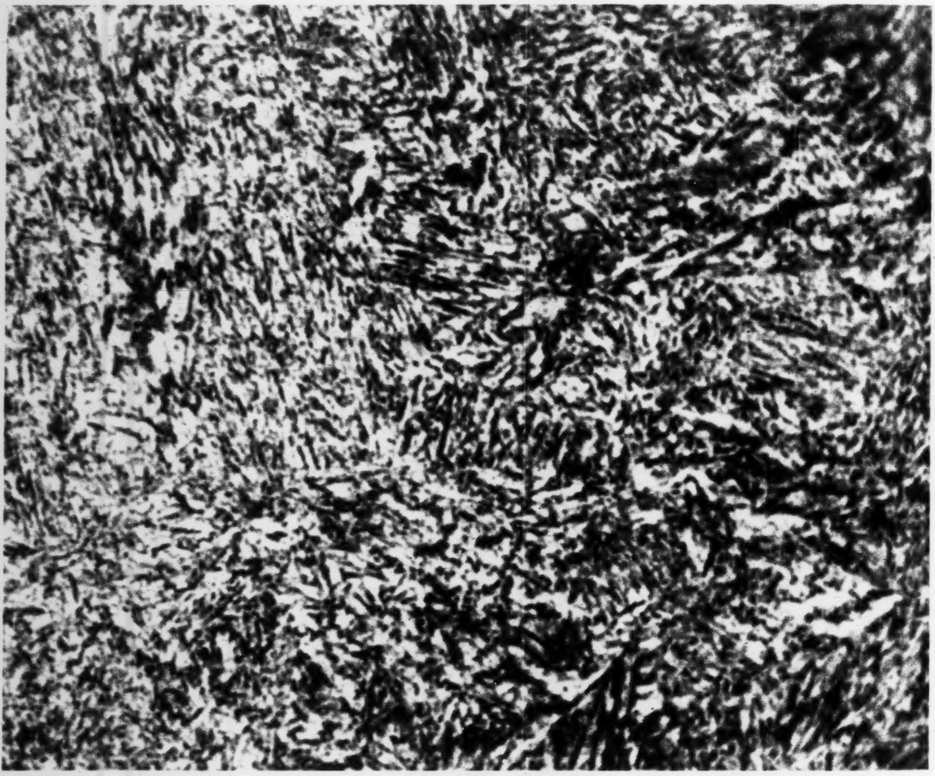

FIG. 33.—Optical Micrograph of Martensite Tempered 1 hr at 500 F (×2500).

## MARTENSITE

*Quenched Martensite:*

The structures resulting from brine quenching from the austenitizing temperature are shown in the micrographs of specimen 50 in Figs. 27 and 28. There is little difference in appearance between martensite in this 4140 steel and in the eutectoid carbon steel described in earlier reports, except that retained austenite was not revealed although X-ray diffraction methods showed the presence of 5 to 6 per cent.

Martensite formed in specimens that were partially transformed prior to quenching appears slightly tempered when compared to those quenched directly. This could be due to a slower cooling rate below the Ms temperature, resulting in more "Q-tempering" as described by Aborn (5).

*Tempered Martensite:*

From the light and electron micrographs of Figs. 29 to 42 it can be seen that the acicular structure of the mar-

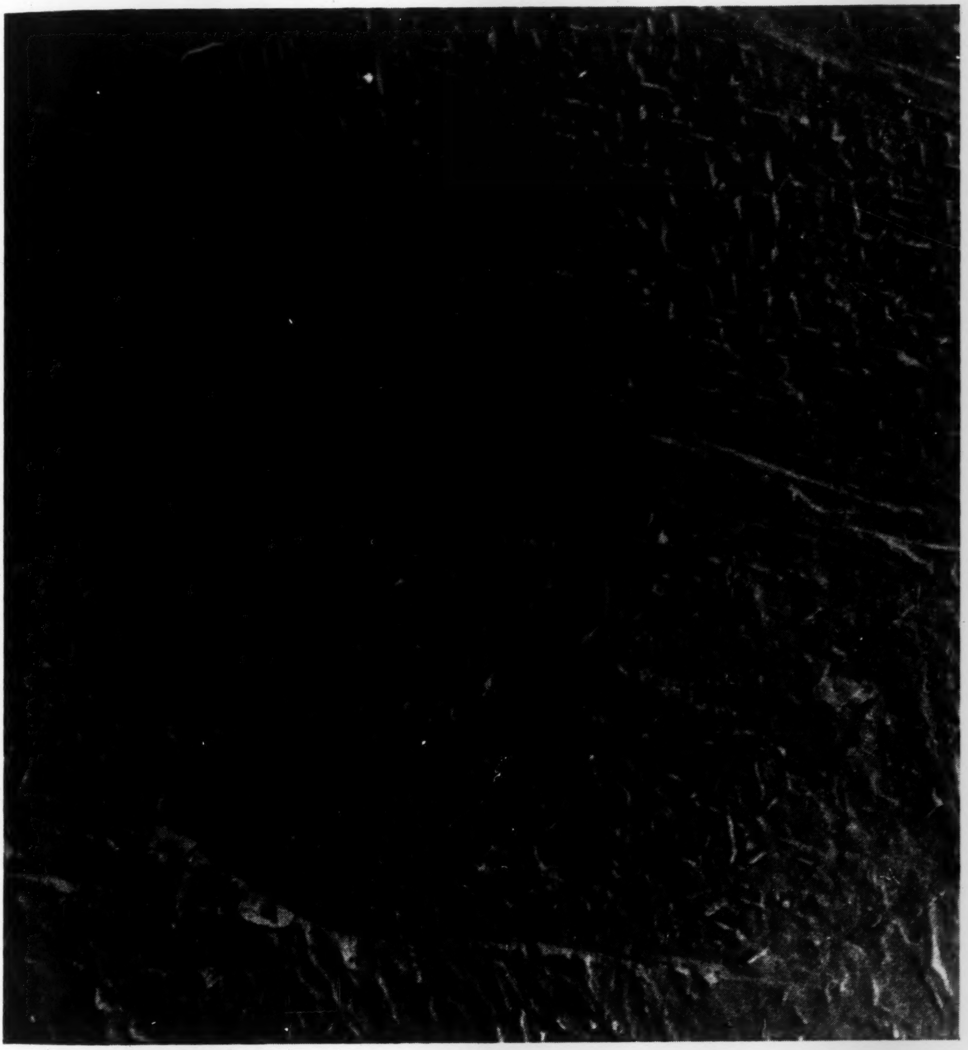

FIG. 34.—Electron Micrograph of Martensite Tempered 1 hr at 500 F (×15,000).

tensite is retained after tempering for 1 hr in the temperature range of 300 to 1300 F. The photomicrographs of specimens 60, 59, and 58 reveal little differentiation between martensite tempered at 300, 400, or even 500 F, but distinct changes are clearly visible in the electron micrographs.

After 1 hr of tempering at 300 F, the original martensite needles are outlined by what appears to be a continuous thin film standing in relief as shown in Fig. 30. These ridges are still present in specimen 59 tempered at 400 F although somewhat discontinuous, as shown in Fig. 32 but are beginning to disappear in specimen 55 tempered at 500 F. The significance of these slow-etching ridges in outlining martensite plates is not fully understood at present, but it is conceivable that they represent films of carbide along the boundary or result from carbon segregation during breakdown of martensite.

Tempering at 300 F produces discrete particles of carbide within the martensite plates similar in appearance to those identified as epsilon carbide by Austin and Schwartz (6) and Teague and Ross (7) in martensite tempered in this temperature range. There is little apparent change in the size of these carbide particles as the tempering temperature is increased from 300 F to 500 F (Fig. 34). Former austenite grain boundaries can be seen in both Figs. 30 and 32.

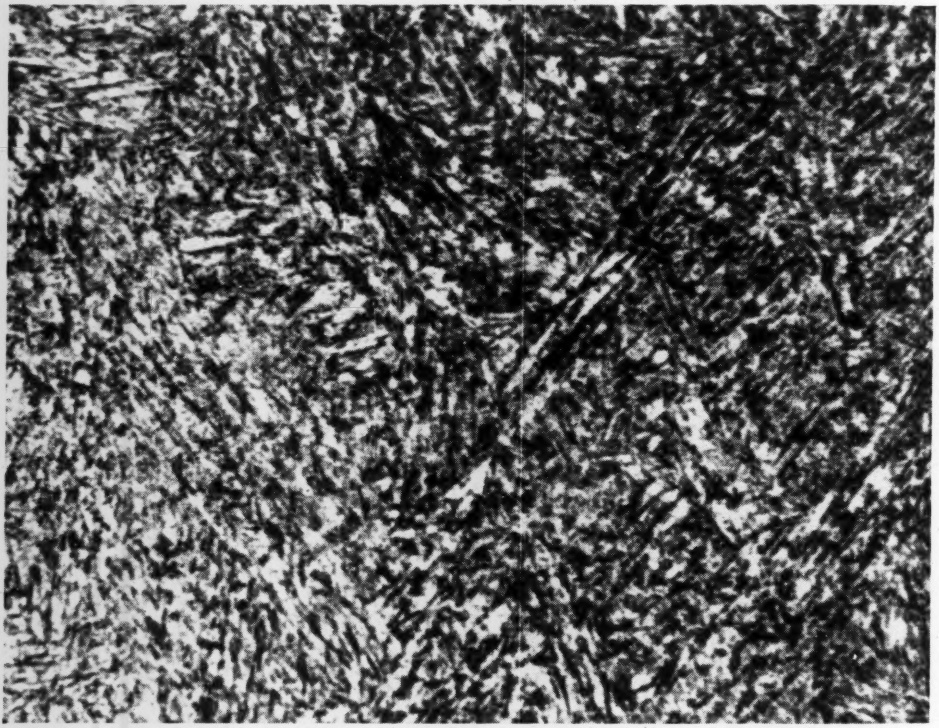

FIG. 35.—Optical Micrograph of Martensite Tempered 1 hr at 700 F (×2500).

Tempering for 1 hr at 700 F results in an electron microstructure containing cementite particles in a ferrite matrix,

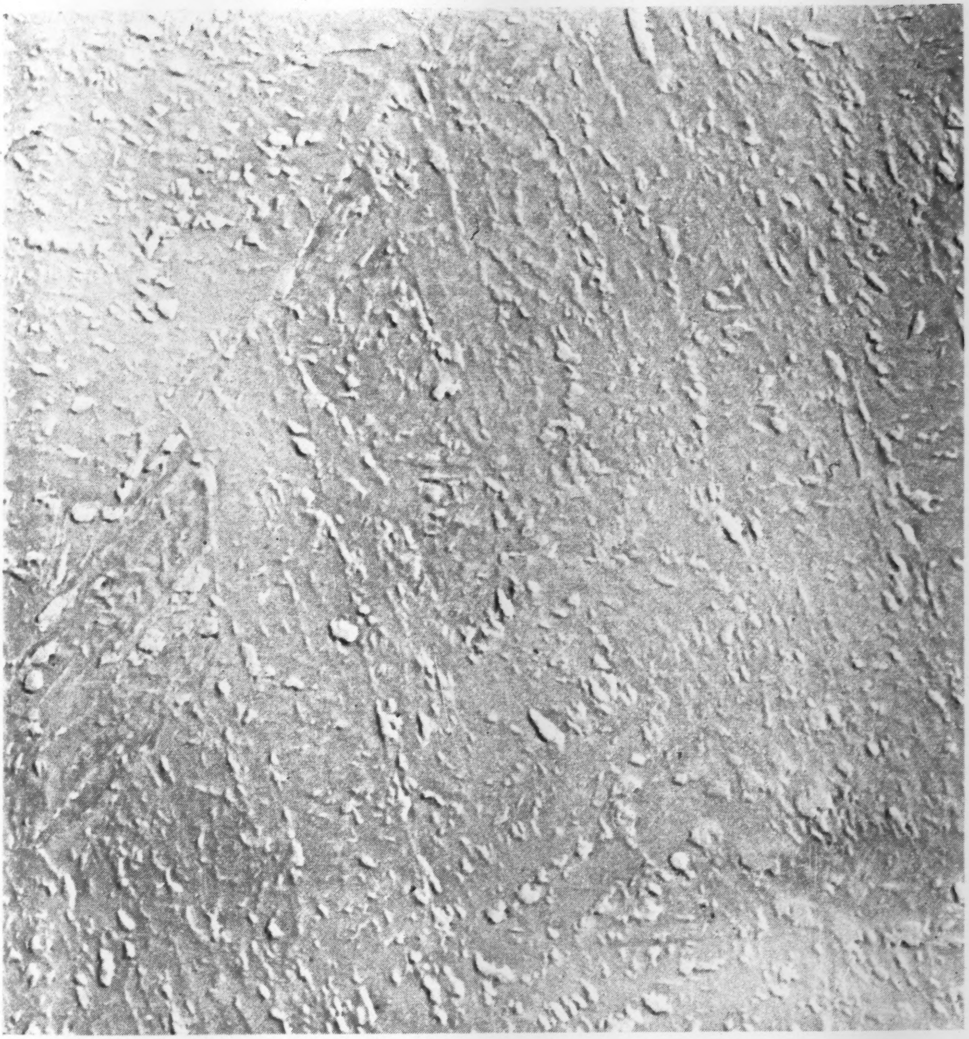

FIG. 36.—Electron Micrograph of Martensite Tempered 1 hr at 700 F (×15,000).

as seen in Fig. 36. No apparent difference is noted between this ferrite and the supersaturated ferrite in the electron micrographs of structures tempered below 400 F. The films surrounding former martensite needles appear to have been supplanted by discontinuous and comparatively elongated particles of cementite, while the fine epsilon carbide particles are not seen. Cementite particles are visible within the former martensite needles in all specimens tempered from 700 F to 1100 F. They grow and become more spheroidized as the temperature of tempering increases, as illustrated by Figs. 38 and 40 showing specimens 53 and 54 tempered at 900 F and 1100 F, respectively. Growth of cementite particles appears most pronounced in the regions of the old martensite boundaries.

Figures 41 and 42 show light and electron micrographs of martensite tempered for 1 hr at 1300 F. The formerly acicular structure of martensite is apparent only in comparison of differently oriented ferrite areas. Spheroidization of cementite is pronounced, but the randomly distributed particles are considerably smaller than in the eutectoid carbon steel.

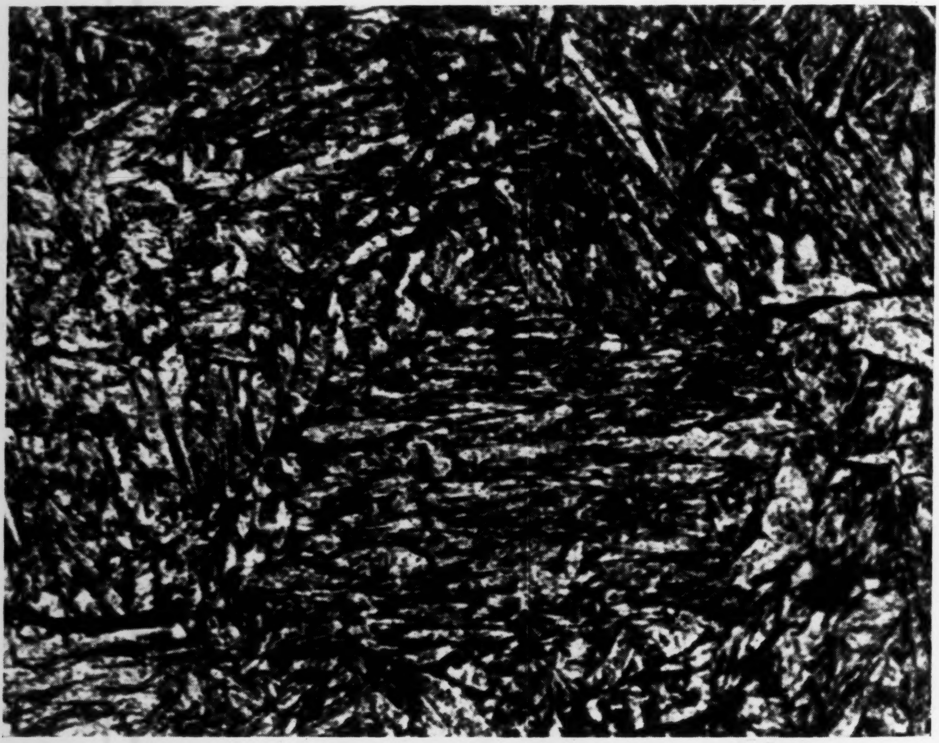

FIG. 37.—Optical Micrograph of Martensite Tempered 1 hr at 900 F (×2500).

## DISCUSSION AND CONCLUSIONS

The results of this work confirm those given in the first report of this Subcommittee on the following points:

1. Similarity of the light and electron micrographs within the limit of the

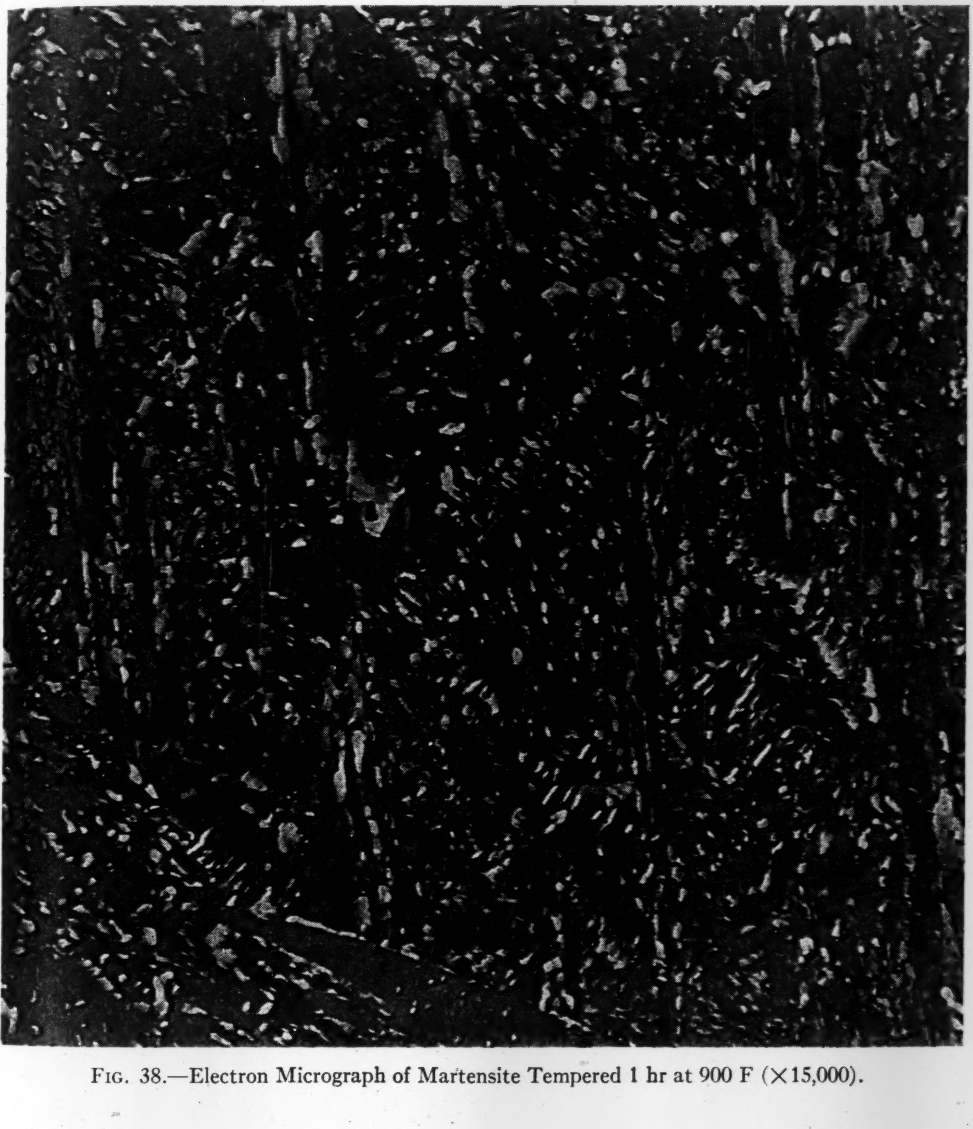

FIG. 38.—Electron Micrograph of Martensite Tempered 1 hr at 900 F (×15,000).

resolving power of the former instrument.

2. Advantage of the much greater resolving power of the electron microscope for the studies of the finer transformation and tempering structures of steels.

In addition, the subcommittee found that the electron metallographic techniques used on the eutectoid carbon steel could be applied to a medium-carbon, medium-alloy steel. There were some differences noted in structures resulting from comparable heat treatment of the two steels but, in general, the interpretation of the micrographs was in substantial agreement. A general shifting to higher temperatures was observed for the isothermal transformation curves for the 4140 steel, and this shift is reflected in the difference in transformation temperature at which comparable electron as well as light microstructures occur in the two types of steel.

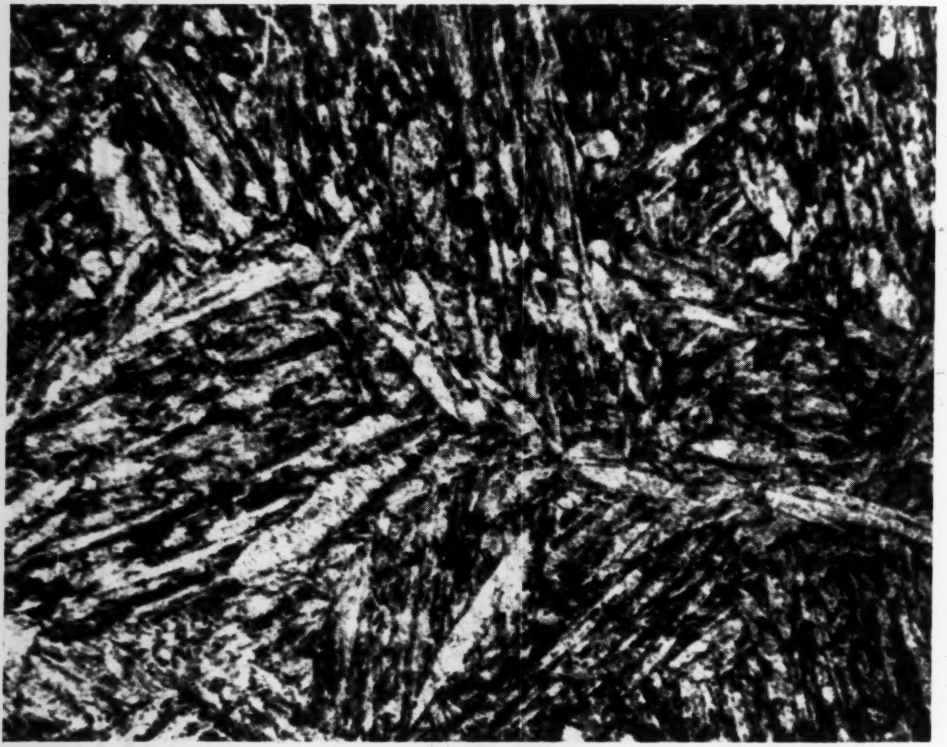

FIG. 39.—Optical Micrograph of Martensite Tempered 1 hr at 1100 F (×2500).

Apparent films around needles of martensite tempered at 300 F to 500 F were observed by Ross, Sernka, and Jominy (8) in AISI 4140 steel as well as in five other medium-carbon, medium-alloy steels. They were described as "percarbide films." Similar films were de-

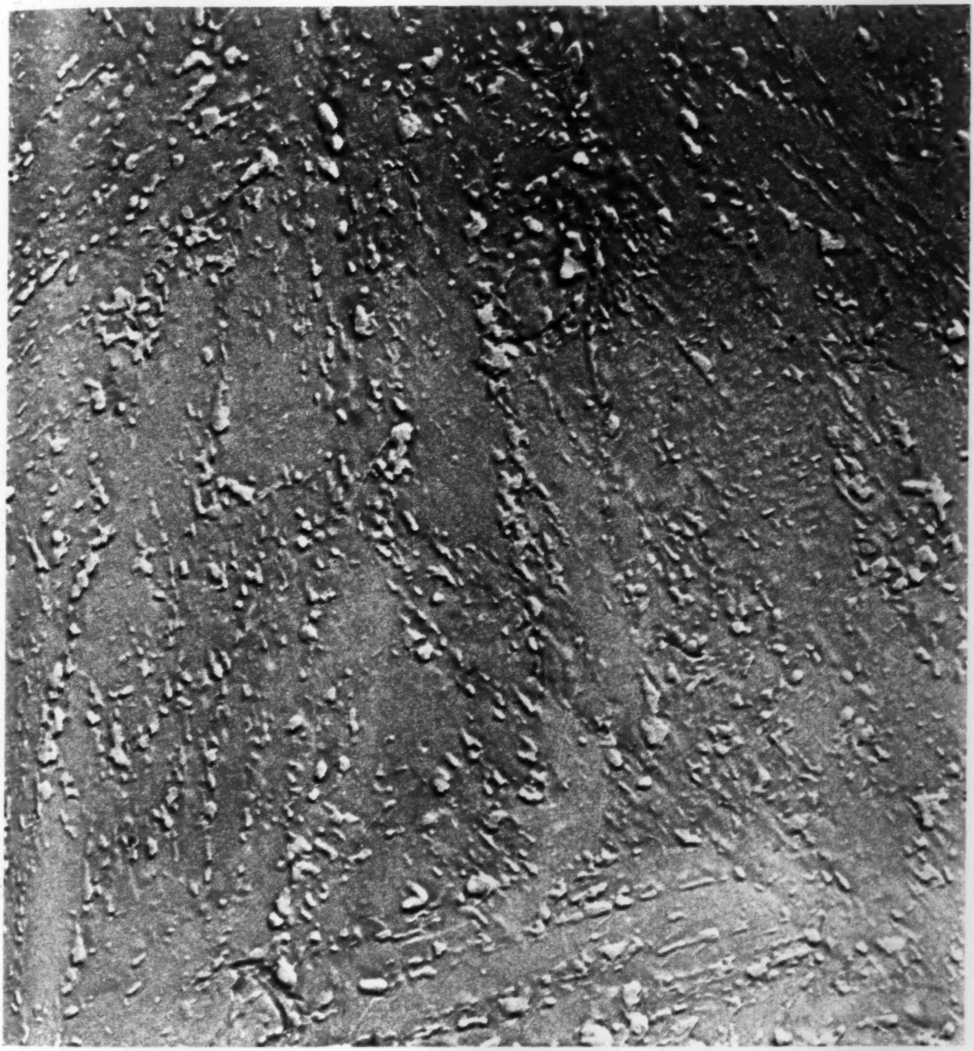

FIG. 40.—Electron Micrograph of Martensite Tempered 1 hr at 1100 F (×15,000).

scribed by Teague and Ross (7) as occurring in eutectoid steel, tempered for 1 hr at temperatures from 200 F to 500 F. Lement, Averbach, and Cohn (9), on the other hand, reported the occurrence of films of cementite between such needles after tempering a series of iron-carbon alloys for 1 hr at temperatures higher than 500 F. They also reported that films of epsilon carbide were found in "martensite sub-boundaries" at tempering temperatures below 450 F.

Consideration of Figs. 30, 32, and 34 indicates that, in AISI 4140 steel, there are no "martensite sub-boundary films"; instead, discrete particles of epsilon carbide appear within the needles as a result of tempering for 1 hr below 500 F. The apparent films seen in these electron micrographs are of such an extent that they envelop entire martensite needles. Figures 36 and 38 show that no films of cementite are formed as the result of tempering AISI 4140 steel above 500 F. They indicate that these martensite needle boundaries are outlined by discrete and elongated particles of cementite which spheroidize as the temperature of tempering increases.

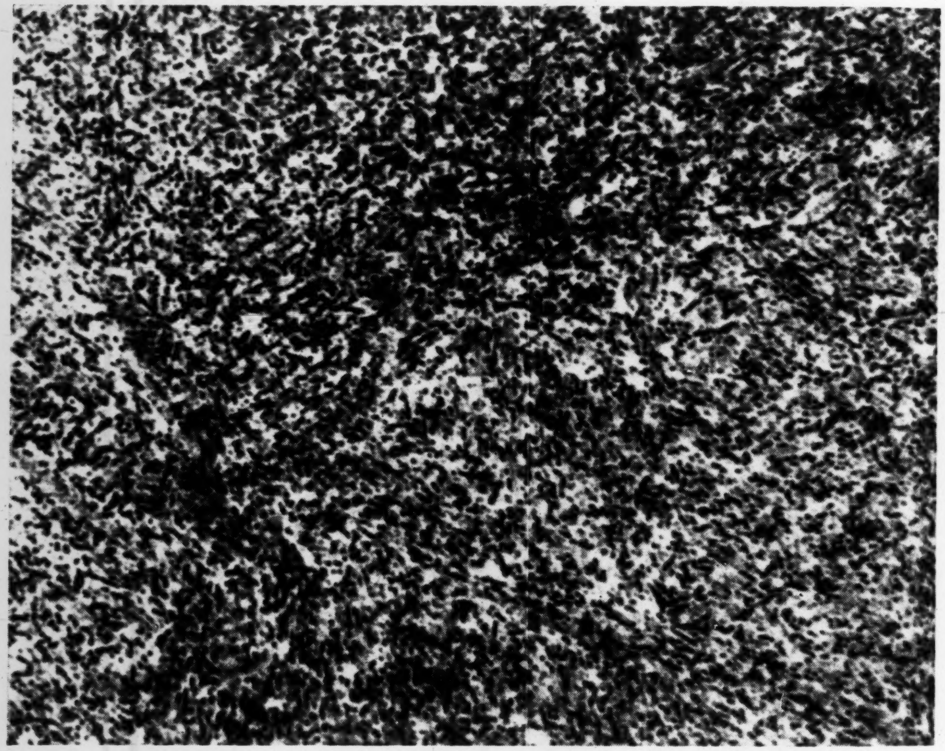

FIG. 41.—Optical Micrograph of Martensite Tempered 1 hr at 1300 F (×2500).

The matrix of martensite tempered above 500 F is ferrite. Martensite tempered below 500 F is considered to have a matrix of supersaturated ferrite. However, distinction between saturated ferrite and supersaturated ferrite is not obtained in the electron micrographs presented in this report.

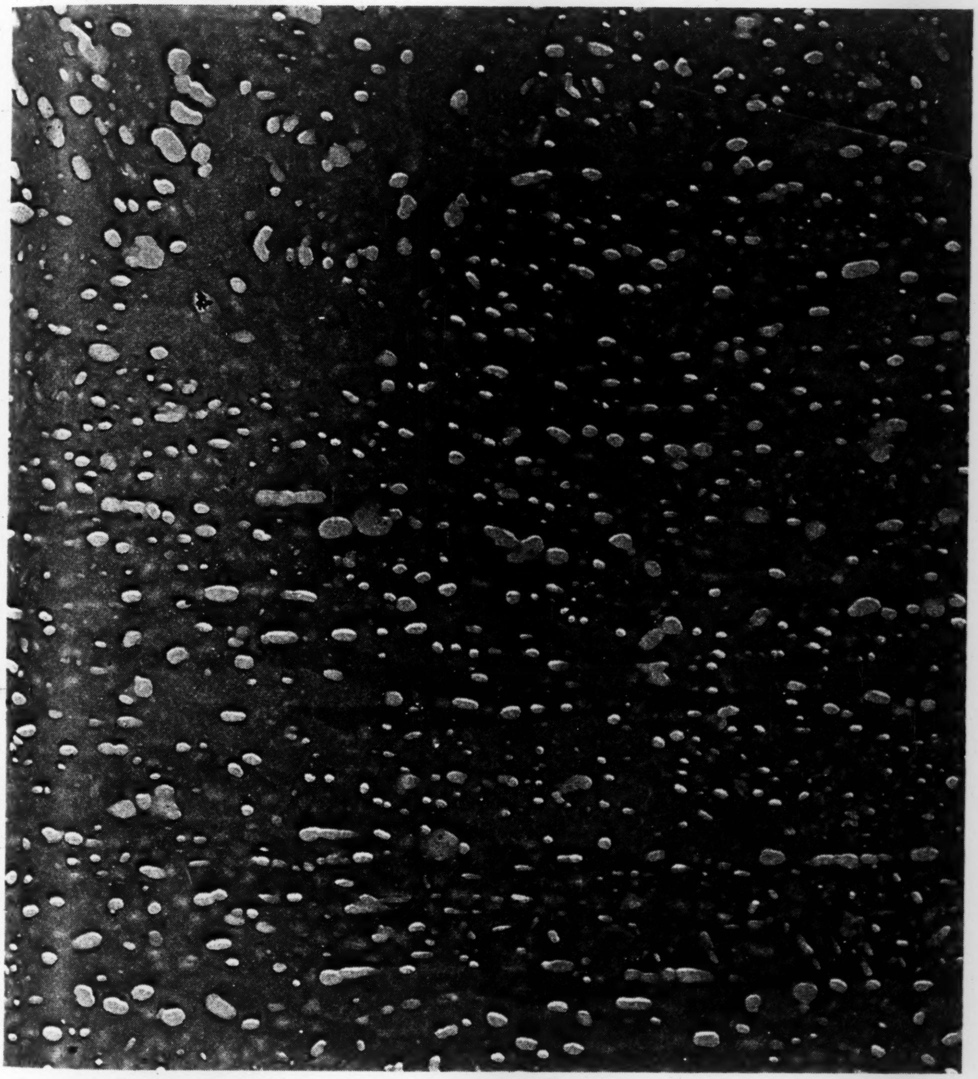

FIG. 42.—Electron Micrograph of Martensite Tempered 1 hr at 1300 F (×15,000).

## REFERENCES

(**1**) "Electron Microstructure of Steel," First Progress Report of Subcommittee XI, ASTM Committee E-4 on Metallography, *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 444 (1950).

(**2**) "Electron Microstructure of Bainite in Steel," Second Progress Report of Subcommittee XI, ASTM Committee E-4 on Metallography, *Proceedings*, Am. Soc. Testing Mats., Vol. 52, pp. 543–571 (1952).

(**3**) "Techniques for Ferrous Electron Metallography," Third Progress Report of Subcommittee XI, ASTM Committee E-4 on Metallography, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, pp. 505–530 (1953).

(**4**) "Electron Microstructure of Tempered Bainite and Tempered Martensite in Steel," Fourth Progress Report of Subcommittee XI, ASTM Committee E-4 on Metallography, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, pp. 569–590 (1954).

(**5**) R. H. Aborn, "Low Carbon Martensites," 1955 Campbell Memorial Lecture, *Transactions*, Am. Soc. Metals, Vol. 48, pp. 51-85 (1956).

(**6**) A. E. Austin and C. M. Schwartz, "Electron Diffraction Study of Iron Carbides in Bainite and Tempered Martensite," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 592 (1952).

(**7**) D. M. Teague and S. T. Ross, "Electron Microstructures of Bainite and Tempered Martensite in Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 590 (1955).

(**8**) S. T. Ross, R. P. Sernka and W. E. Jominy, "Some Relationships between Endurance Limit and Torsional Properties of Steel," *Transactions*, Am. Soc. Metals, Vol. 48, pp. 119–139 (1956).

(**9**) B. S. Lement, B. L. Averbach and M. Cohen, "Microstructural Changes on Tempering Iron-Carbon Alloys," *Transactions*, Am. Soc. Metals, Vol. 46, pp. 851–877 (1954).

## APPENDIX III

# OBSERVATIONS ON THE DISLOCATION STRUCTURE OF SUB-BOUNDARIES IN IRON* †

By F. W. C. Boswell[1]

In recent years several workers have reported etching techniques whereby pits can be formed at individual dislocation sites in crystals. No one method is of general application, but suitable etching procedures have been developed for germanium (**1**),[2] silicon (**2**), some alkali halides (**3, 4**), and a small number of metals and alloys (**5, 6, 7**). These techniques have proved useful in the study of crystal perfection and the experimental investigation of the motions and interactions of dislocations during plastic deformation and annealing.

Recent work by the author has indicated that, under certain conditions, etch pits can be developed at edge dislocation sites in high purity iron. The present paper outlines the technique used and reports some observations on the characteristics and distribution of these pits along sub-boundaries.

## EXPERIMENTAL

The observations reported here were made on vacuum-melted iron obtained from the Vacuum Metals Corp. of Cambridge, Mass. The analysis in per cent by weight was as follows: carbon $<0.01$, oxygen 0.004, nitrogen 0.001, nickel 0.03, silicon 0.007, hydrogen $<0.005$, sulfur $<0.003$, other impurities 0.001 per cent.

Specimens used in this work were extended a few per cent at room temperature, annealed at 850 C in dry hydrogen for 15 hr and furnace cooled. The grains were a few millimeters in diameter. The slow cooling from an elevated temperature appears to be a necessary prerequisite for the development of etch pits at dislocations by the method used here. It has recently been reported (**8**) that etch pit formation at dislocations in iron-silicon alloys is critically dependent on carbon segregation. It appears probable that a somewhat similar effect occurs in high purity iron and that the pit formation is hence dependent on both heat treatment and carbon content.

Surfaces were electropolished by a method previously described (**9**) and then etched as follows:

(1) etch in 1 per cent nitric acid in ethanol for 1 min,

(2) rinse in methanol,

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* Presented at a meeting of Subcommittee XI on Electron Microstructure of Metals of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 18, 1957.

† Published by permission of the Director, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Ont. (Canada).

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 526.

(3) etch in freshly made 0.5 per cent picric acid in methanol for 5 min, and

(4) rinse and dry.

Replicas for electron microscopy were of Parlodion backed with Elvanol. Shadowing was carried out with chromium at an angle $\tan^{-1} \frac{1}{4}$ to the plane of the replica.

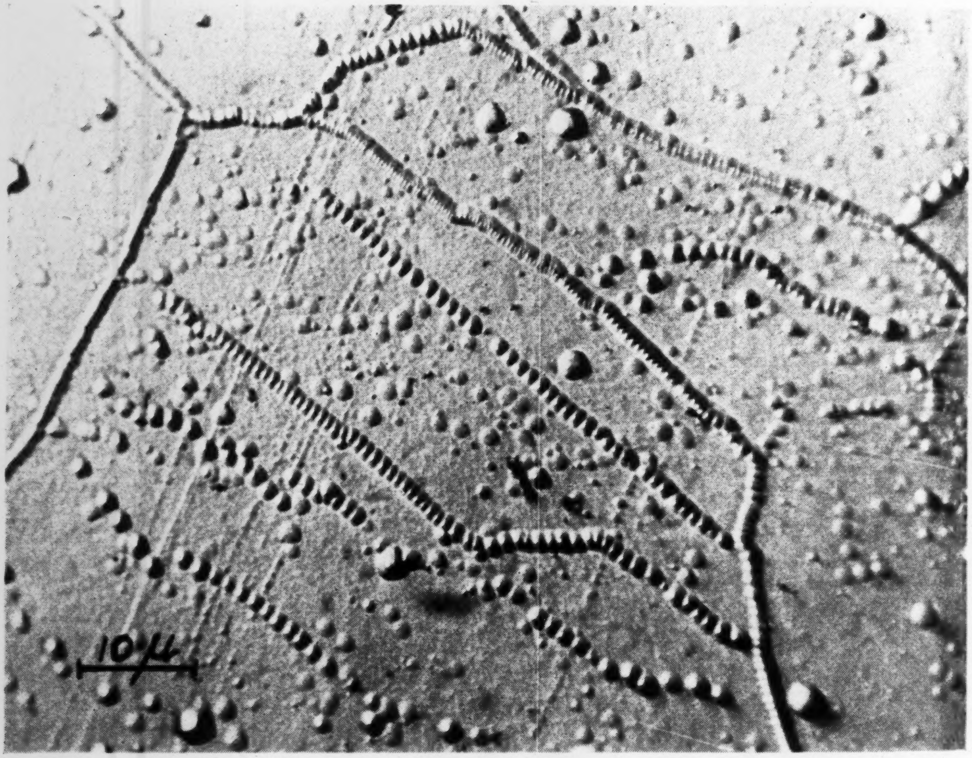

FIG. 1.—Light Micrograph of Sub-Boundaries in High-Purity Iron.

## RESULTS AND DISCUSSION

Figure 1 is a light micrograph showing sub-boundaries observed in a small percentage of the grains of the recrystallized iron specimens. This substructure is believed to have formed as a result of some slight deformation at the elevated temperature subsequent to the recrystallization of the particular grain observed. These sub-boundaries are closely related to the well-known veining structure in ferrite. Several of the sub-boundaries in Fig. 1 are clearly resolved as rows of regularly spaced dots similar in appearance to the rows of dislocation etch pits reported in other materials (1–8). The author has carried out experiments on bent and polygonized single crystals of iron by which it has been shown that the dots produced in iron by the etching technique used here correspond to the sites of individual edge dislocations. These bend experiments will be reported in more detail elsewhere.

A shadowed electron micrograph of sub-boundaries in a polycrystalline specimen similar to that of Fig. 1 is shown in Fig. 2. It may be noted that the densities of pits along three boundaries meeting at a point are in qualitative agreement with theoretical predictions. From the electron micrograph it is clear that the dots observed in Fig. 1 are actually pits and the shape of the pits, which is of theoretical interest, can be deduced. Each pit consists of a shallow, saucer-shaped depression about 10,000 Å across with a much sharper depression about 1000 Å across and a few hundred Å deep toward the center of the shallow depression. It appears possible that the extended shallow region is associated with the stress field of the dislocation while the more severe attack at the core is associated with solute segregation. It has been noted that in some cases the pits are symmetrical with the deeper pit centered in the shallow depression,

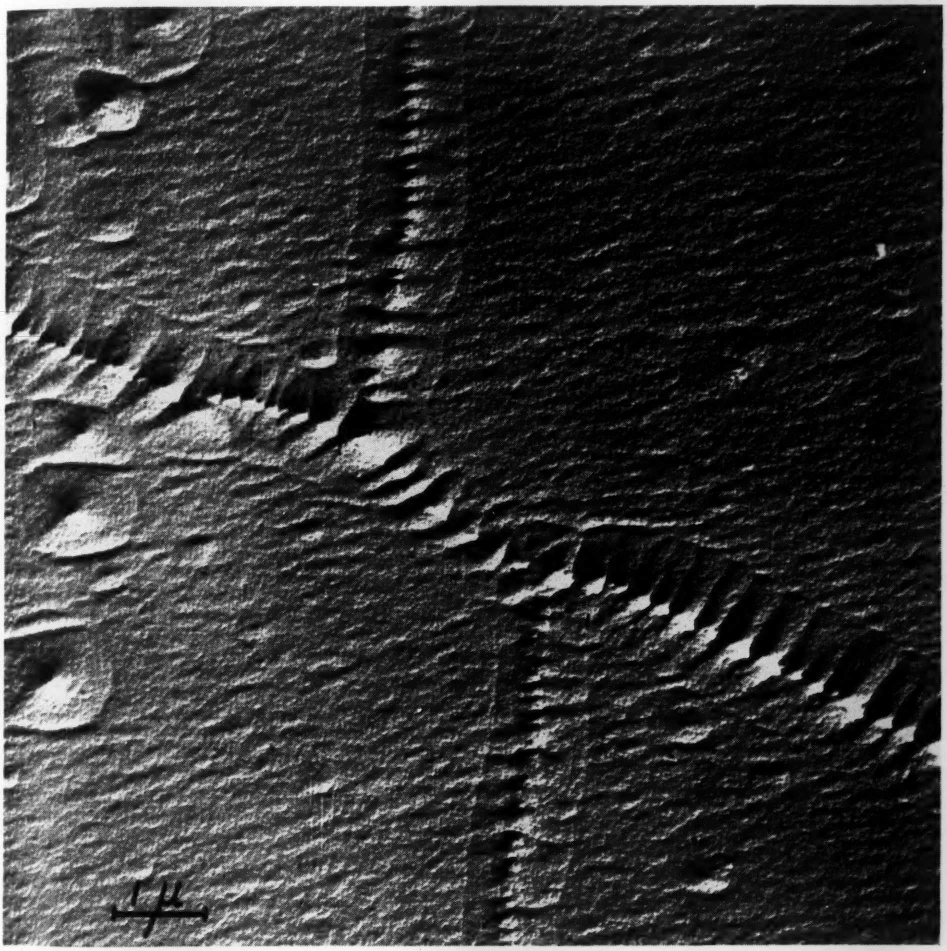

FIG. 2.—Chromium-Shadowed Electron Micrograph of Sub-Boundaries in High-Purity Iron.

whereas in other cases the pits are markedly asymmetrical with the deeper pit displaced toward one edge of the saucer. From the fact that both kinds of pits are often observed in the same field, as in Fig. 3, it appears that the asymmetry depends on the angle at which the dislocation line intersects the surface. This fact may prove useful in the determination of the orientation of dislocation lines.

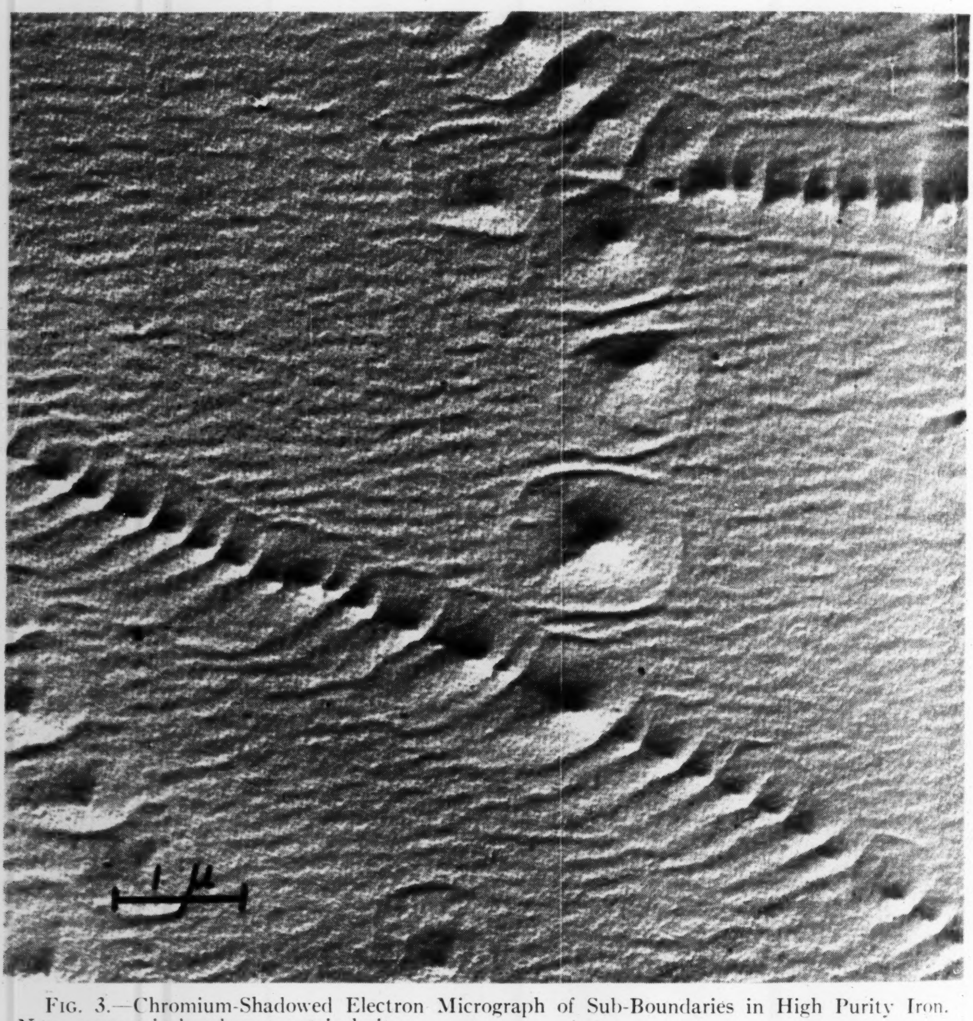

FIG. 3.—Chromium-Shadowed Electron Micrograph of Sub-Boundaries in High Purity Iron. Note symmetrical and asymmetrical pits.

Since the etching mechanism involved in these experiments produces pits at dislocation sites, it appears to be entirely different from that recently reported by F. van Wijk and J. A. B. van Dijck (10). These workers observed regularly-spaced protruding etch marks in electron micrographs of iron etched by an electrolytic method and they suggested that the protrusions indicated the sites of dislocations. Another marked difference in our results and those of van Wijk and van Dijck is that we observed only a few scattered pits on the surface, apart from those in the sub-boundaries, while the above authors observed the whole surface to be covered with closely spaced etch marks so that only a small proportion was contained in the sub-boundaries.

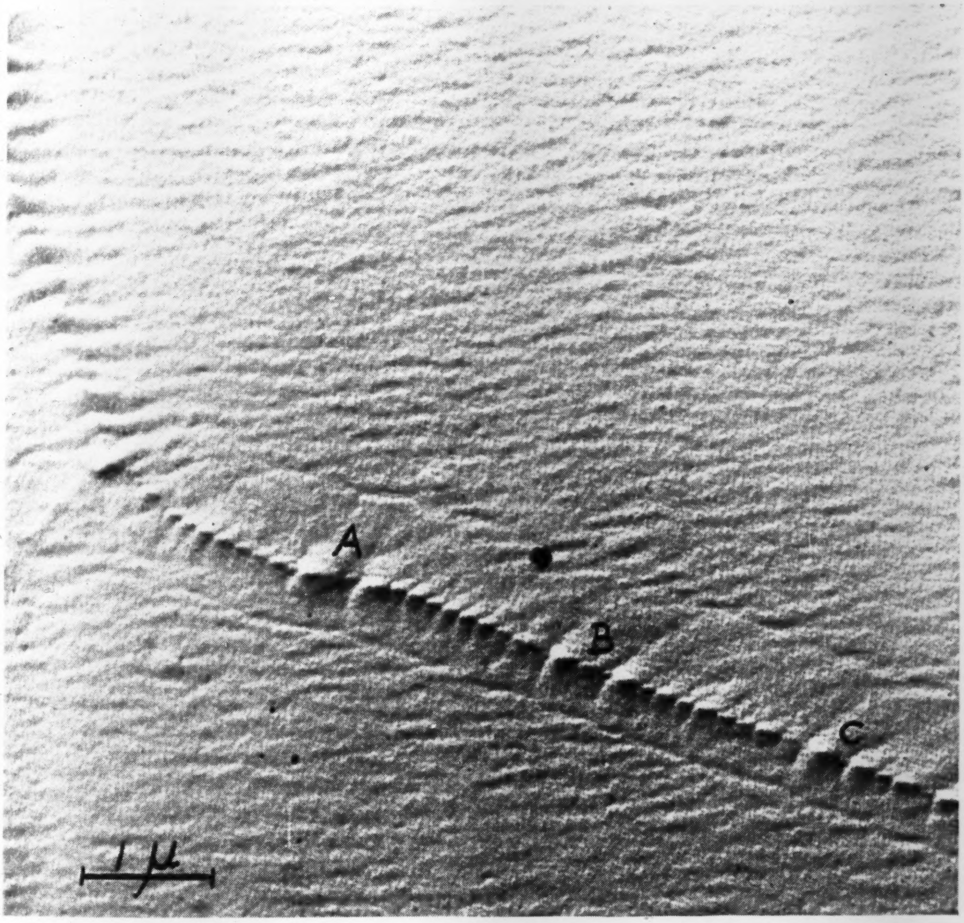

FIG. 4.—Chromium-Shadowed Electron Micrograph of Sub-Boundaries in Alpha Iron. Note periodic interruption in regular spacing along the lower boundary at points *A*, *B*, *C*.

The resolution of the etching process is below that of the light microscope. For instance, in the lower boundary of Fig. 4 the clearly resolved pits are 0.15 μ apart. This dislocation spacing corresponds to a boundary tilt of about 12 min. Thus electron microscopy is the preferred method for studying the structure of the sub-boundaries.

There is no definite evidence from our observations that there is any tendency for pits to form at screw dislocations.

However, in some cases a repeated interruption in the regular spacing along a sub-boundary has been noted. This effect can be seen in Fig. 4. It apparently arises from the occurrence along the sub-boundary of dislocations whose etching characteristics are slightly different from those of the majority of the dislocations in the boundary. At present we do not know whether this effect is associated with the occurrence of screw dislocations or whether the etching characteristics of edge dislocations lying in differing slip planes would vary sufficiently to cause the variation. Further work on single crystals should help to clarify this point.

## CONCLUSIONS

1. Tilt boundaries in iron, of angles up to about 12 min, can be studied in terms of their dislocation structure by means of a suitable etching technique combined with electron microscopy.

2. Structural features of theoretical interest have been observed in low angle boundaries in iron.

3. These techniques should be useful in the experimental investigation of sub-boundary formation in iron, in studying phase transformations at sub-boundaries in relation to the dislocation structure of the sub-boundary, and in relating nucleation in surface reactions on iron to dislocation sites in the surface.

## REFERENCES

(1) F. L. Vogel, "Dislocations in Low-Angle Boundaries in Germanium," *Acta Metallurgica*, Vol. 3, p. 245 (1955).

(2) F. L. Vogel and L. Clarence Lovell, "Dislocation Etch Pits in Silicon Crystals," *Journal of Applied Physics*, Vol. 27, p. 1413 (1956).

(3) S. Amelinckx, "Etchpits and Dislocations Along Grain Boundaries, Sliplines and Polygonization Walls," *Acta Metallurgica*, Vol. 2, p. 848 (1954).

(4) J. J. Gilman and W. G. Johnston, "Observations of Dislocation Glide and Climb in Lithium Fluoride Crystals," *Journal of Applied Physics*, Vol. 27, p. 1018 (1956).

(5) W. R. Hibbard, Jr., and C. G. Dunn, "A Study of (112) Edge Dislocations in Bent Silicon-Iron Single Crystals," *Acta Metallurgica*, Vol. 4, p. 306 (1956).

(6) K. T. Aust and R. Maddin, "Subgrain and Electrical Resistivity Studies of Molybdenum Single Crystals," *Acta Metallurgica*, Vol. 4, p. 632 (1956).

(7) J. J. Gillman, "Structure and Polygonization of Bent Zinc Monocrystals," *Acta Metallurgica*, Vol. 3, p. 277 (1955).

(8) J. C. Suits and J. R. Low, "Dislocation Etch Pits in Silicon Iron," *Acta Metallurgica*, Vol. 5, p. 285 (1957).

(9) F. W. Boswell, "Electropolishing of Ferrous Metal Specimens for Electron Metallography," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 426 (1956).

(10) F. van Wijk and J. A. B. van Dijck, "Investigation of Pure Iron and Soft Steel With the Electron Microscope," *Acta Metallurgica*, Vol. 4, p. 657 (1956).

## APPENDIX IV

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# THE PREPARATION OF SHADOWED PLASTIC REPLICAS*

### BY S. T. ROSS[1] AND D. M. TEAGUE[2]

Although a number of different procedures have been utilized for reproducing the microstructures of metal surfaces, the shadowed negative plastic replica has been most commonly used. This is due to its being rapid, fairly simple, and capable of reproducing the majority of structures of interest (**1**).[3] Through experience, it has been possible to develop procedures which permit magnifications up to 30,000×, with a resolving power better than 100 Å, without the introduction of extraneous replica structure.

The shadowing of the plastic replica is an important step in its preparation, since shadowing enhances the contrast and detail. Figure 1 shows the appearance of a plastic replica which has not been metal shadowed. Only the gross details are visible. Studies of shadowing techniques and materials (**2**) have revealed that several metals provide excellent results, including chromium, uranium, palladium-platinum and others. A relatively high shadow angle, about 45 deg to the angle of the surface, produces good detail in coarse structures without excessive highlights and apparent shadows. For very fine microstructures, a shadow angle of about 30 deg to the surface is preferable.

Figure 2 is an electron micrograph of the identical specimen shown in Fig. 1. This replica has been shadowed with chromium, and now reveals the very fine epsilon iron carbide particles which are present in this structure (**3**).

After a photographic negative has been prepared of the shadowed plastic replica in the electron microscope, a direct photographic print produces an appearance which reproduces the topography of the original surface. The over-all result is an image which appears as though the original surface were illuminated with oblique light. This representation requires no special interpretation since it is similar to the result obtained by light microscopy.

For the electron microscopist interested in the study of microstructures, a detailed summary of the procedures used in the preparation of the accompanying micrographs follows.

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* Presented at a meeting of Subcommittee XI on Electron Microstructure of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 18, 1957.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 535.

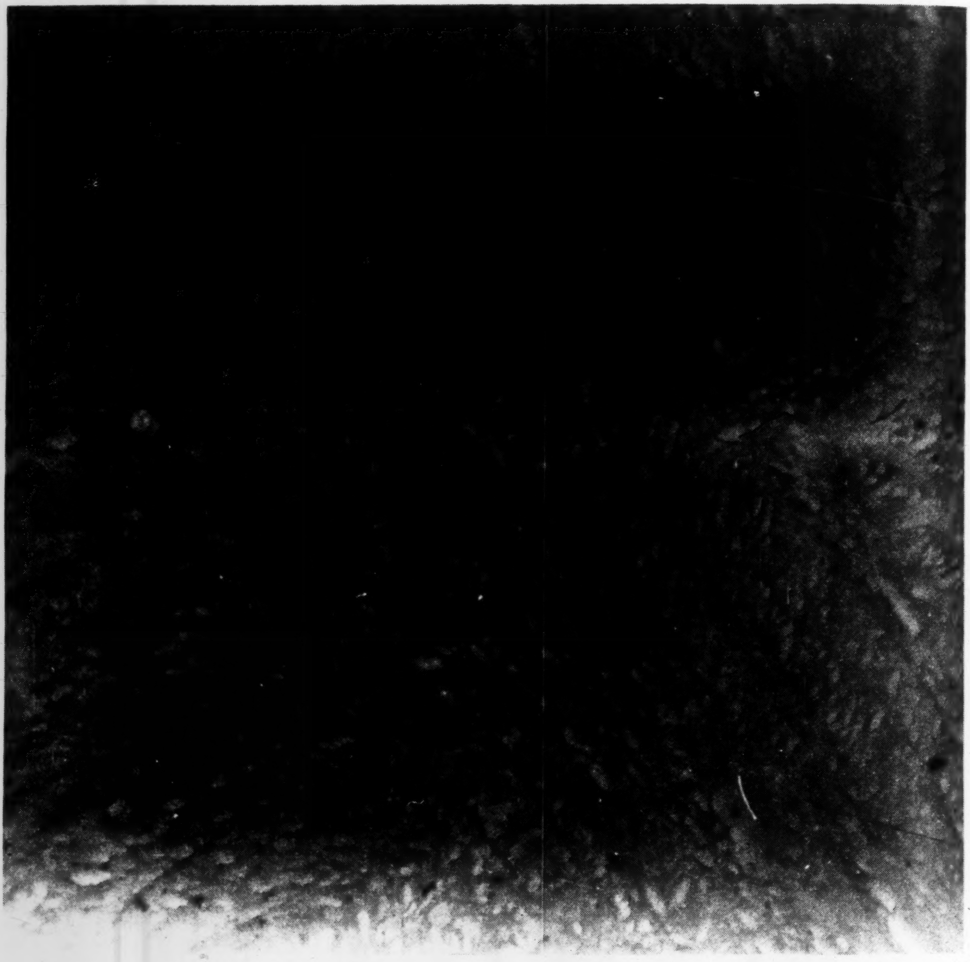

FIG. 1.—Electron Micrograph of Bainite, Isothermally Transformed at 500 F, Picral Etch. Unshadowed Plastic Replica (×15,000).

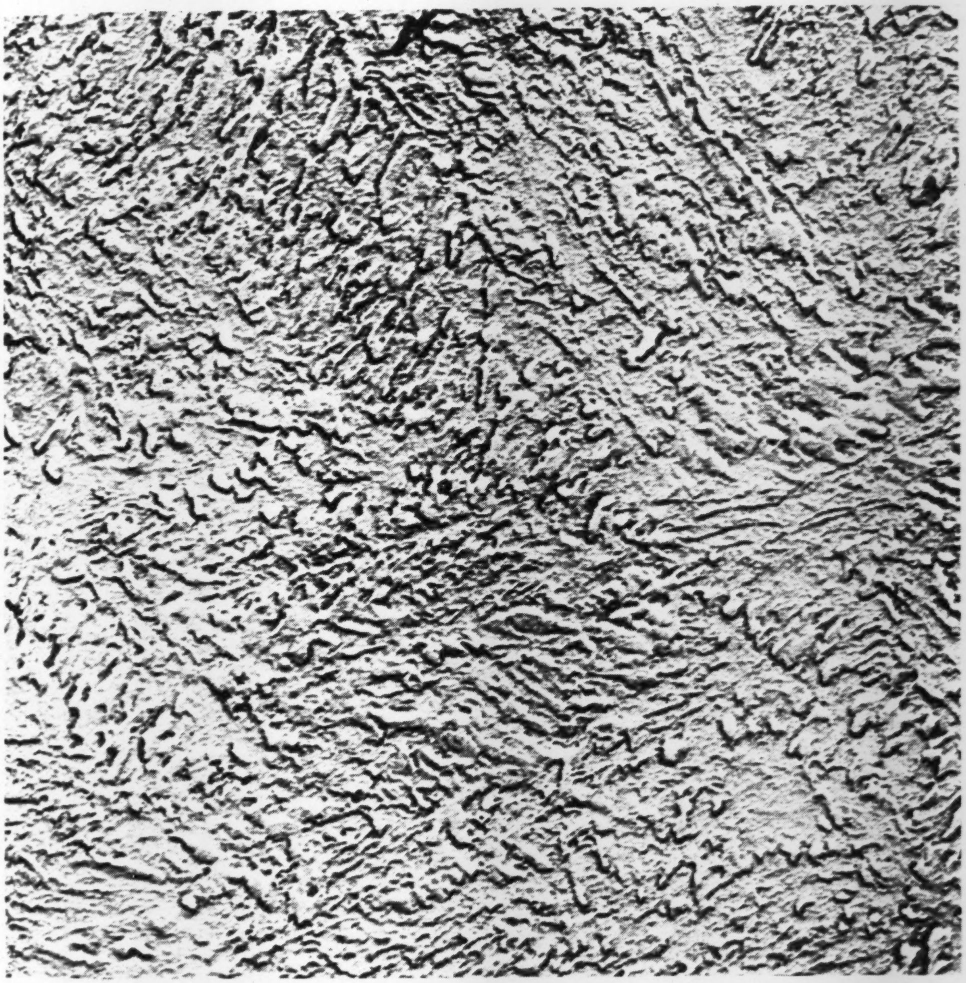

FIG. 2.—Electron Micrograph of Bainite, Isothermally Transformed at 500 F, Picral Etch, Shadowed Plastic Replica (×30,000).

## PROCEDURE FOR THE PREPARATION OF A SHADOWED PLASTIC REPLICA

### (One-Step or Negative Plastic Replica)

After the specimen has been etched, the plastic solution is flowed over the prepared surface and the excess allowed to drain off. The volatile solvent rapidly evaporates, leaving a very thin (300 to 400 Å) plastic film. Typical plastic solutions[4] are:

(1) Parlodion (collodion) dissolved in amyl acetate. A concentration of 0.75 per cent Parlodion is commonly used. More dilute solutions provide greater contrast with very light etches, while higher concentrations are used with heavily etched specimens. Parlodion is a purified pyroxylin and can be obtained from Mallinckrodt Chemical Co. For additional film strength, the Parlodion replica can be backed with 5 to 10 per cent aqueous polyvinyl alcohol, grade 51-05, available from E. I. du Pont de Nemours & Co., Inc.

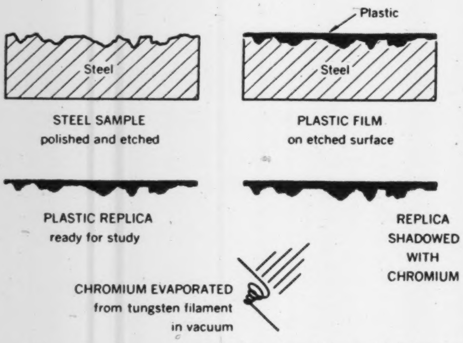

FIG. 3.—Steps in the Preparation of a Shadowed Plastic Replica.

(2) Formvar dissolved in chloroform, dioxane, or ethylene dichloride. A freshly prepared 0.75 to 0.9 per cent solution of Formvar is used. After being in solution for a period of time, the Formvar appears to polymerize to produce a visible structure in the replica film. Medium viscosity Formvar, such as Monsanto sample 15–95, type E, may be used. For rough structures the Formvar can be backed with a 5 per cent solution of Zapon, available in that form from Atlas Powder Co., Stamford, Conn.

The replica film is removed by moistening the surface of the specimen with the breath and then picking it up with cellophane tape upon which a specimen screen has been placed. An alternate method of stripping is to back the replica film with a heavy coating of plastic and then strip the two films together. This procedure avoids strain markings in the replica when stripping from very rough surfaces. Formvar or ethyl cellulose replicas may be backed with Parlodion or Zapon, which can be removed by washing in amyl acetate. A 10 per cent solution of polyvinyl alcohol in water can be used to back collodion or Parlodion replicas for stripping, and then dissolved away in warm water.

Shadowing is usually accomplished by stripping the plastic replica from the specimen, then placing this replica in a vacuum chamber with the face which was stripped from the metal surface facing an evaporation filament or boat. Metal for evaporation is placed in the filament, or boat, which is heated electrically after the chamber has been evacuated. The replica is tilted at an angle to the source of the evaporating metal so that the metal vapor, which travels in a straight line in vacuum, is deposited on the contours of the replica at an oblique angle (Fig. 3). It may be noted that the metal deposit (shadow area) produces a white "highlight" at the edge of a raised portion of the metal surface.

Several metals may be used for

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[4] Specific examples of source and grade, only, are given; this is not a complete listing of suitable materials.

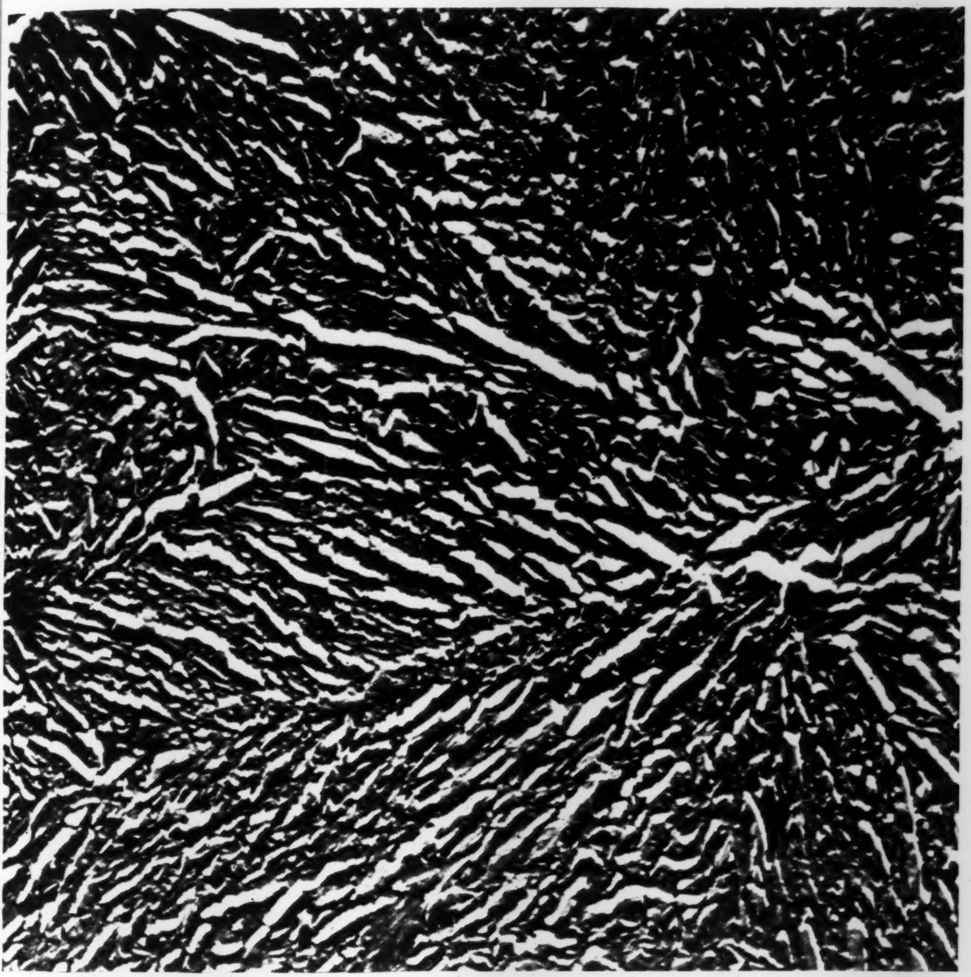

FIG. 4.—Electron Micrograph of Bainite, Isothermally Transformed at 750 F, Picral Etch, Heavily Shadowed Plastic Replica (×15,000).

vacuum shadowing. Chromium is most commonly used, and provides excellent results, although uranium, platinum-palladium and aluminum-gold may be used for greater density.

The metal is deposited at an angle of from 30 to 45 deg to the plane of the replica, depending on the size of the detail in the specimen. A lower angle is preferable for fine detail, whereas the higher angle is used for coarser structures. The length of the highlight and apparent shadow is increased by shadowing at a lower angle. The result of heavily shadowing a replica at a low angle, less than 30 deg, is illustrated in Fig. 4. Some fine detail is lost, and the surface contours are more difficult to visualize.

The detailed effect of shadowing a plastic replica can be described with the

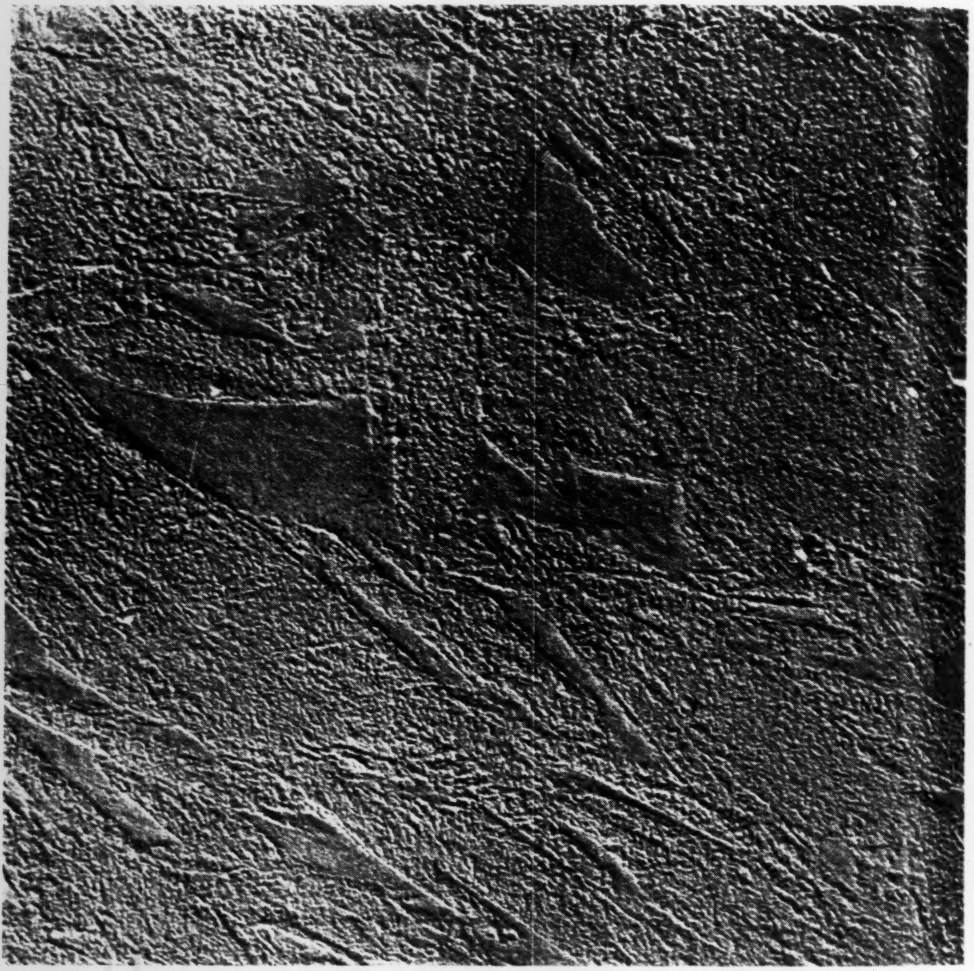

FIG. 5.—Electron Micrograph of Tempered Martensite, 1 hr at 250 F, Picral Etch, Shadowed Plastic Replica (×30,000).

aid of Fig. 3. The metal is evaporated onto the plastic at an angle. This coats the near side of the microscopic hills, while the far sides and valleys are relatively uncoated. When the shadowed replica is placed in the electron microscope, the electron beam is absorbed by the heavier metal layer on the near side of the microscopic hills, producing a white area in the negative plate. When direct prints are made, according to the usual procedure, these light areas appear as dark shadows on the final print. These dark shadows (see Fig. 3) therefore actually correspond to the valleys in the original etched metal surface. The overall result is to produce an electron micrograph with shadows having the same

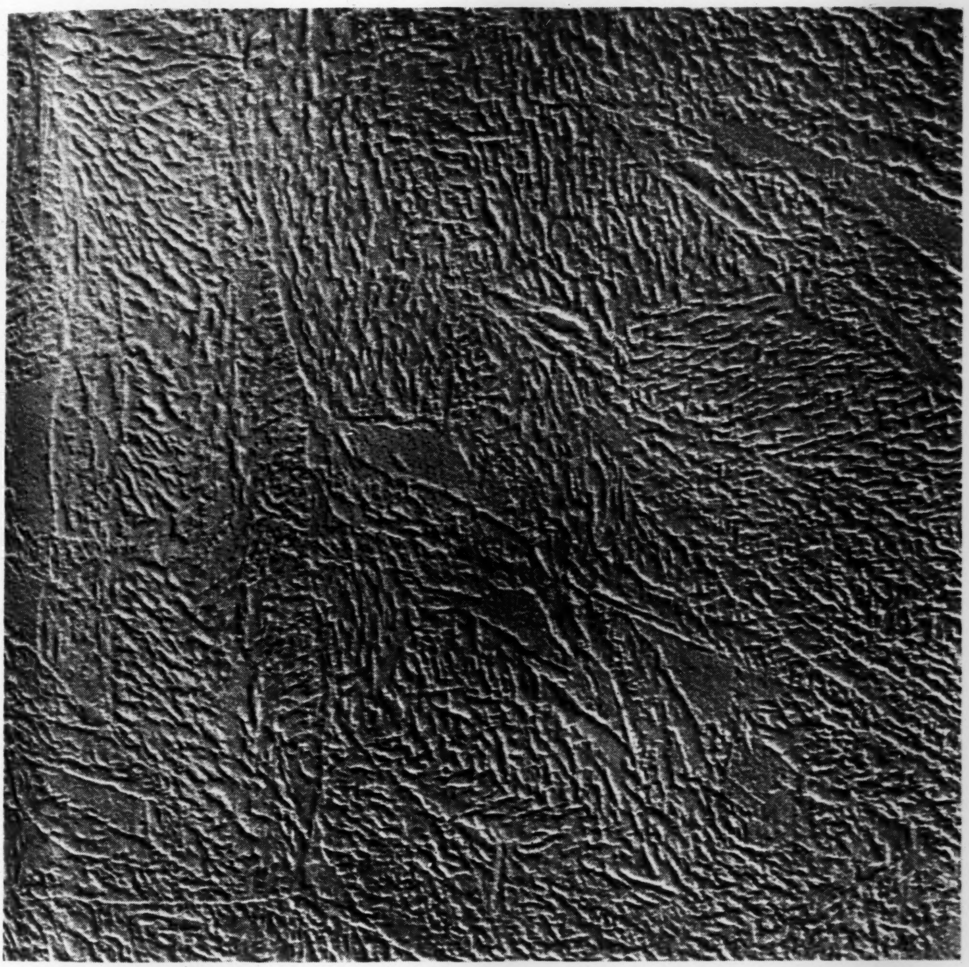

FIG. 6.—Electron Micrograph of Tempered Martensite, 1 hr at 400 F, Picral-Zephiran Chloride Etch, Shadowed Plastic Replica (×30,000).

relative position and appearance as though the original surface were being observed with oblique lighting. Highlights and shadows have a normal appearance and assist in the interpretation of surface microstructures (**2**).

The negative plastic replica can be prepared, stripped, and shadowed with moderate rapidity. This makes it possible to follow the etching process or to study a structure promptly and thoroughly.

Figure 5, eutectoid steel martensite tempered 1 hr at 250 F (**4**), is an illustration of the use of a shadowed plastic technique. It is an illustration of the fine detail which it is possible to obtain. Figure 6 is a micrograph of martensite tempered 1 hr at 400 F. The surface

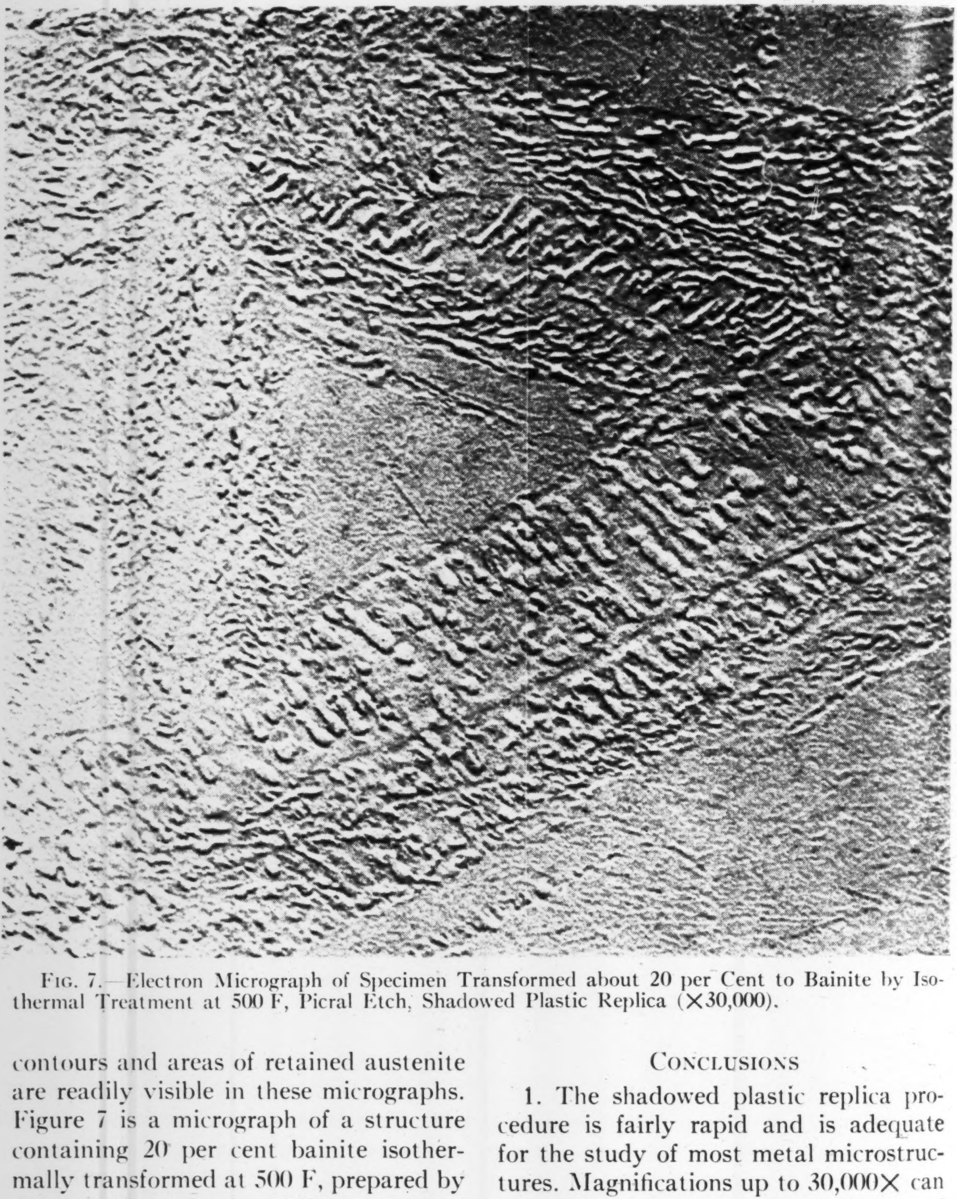

FIG. 7.—Electron Micrograph of Specimen Transformed about 20 per Cent to Bainite by Isothermal Treatment at 500 F, Picral Etch, Shadowed Plastic Replica (×30,000).

contours and areas of retained austenite are readily visible in these micrographs. Figure 7 is a micrograph of a structure containing 20 per cent bainite isothermally transformed at 500 F, prepared by the negative plastic replica procedure (4).

## CONCLUSIONS

1. The shadowed plastic replica procedure is fairly rapid and is adequate for the study of most metal microstructures. Magnifications up to 30,000× can be utilized without the interference of the structure of the replica itself.

2. Direct printing of photographic negative from the shadowed plastic replica produces a micrograph which has the same reproduction of surface contour as though one were viewing the structure directly. This is convenient from the standpoint of photographic processing and ease of interpretation.

*Acknowledgments*:

We would like to express appreciation to Mr. George E. Pellissier for assistance in preparing this paper, and to Mr. F. O. Thomas for several of the electron micrographs.

## REFERENCES

(1) "Techniques for Ferrous Electron Metallography," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 505 (1953).

(2) D. M. Teague, "Metal Shadowing for Contrast Enhancement—Comparison of Shadow Metal and Shadow Angle," Symposium on Techniques for Electron Metallography, Am. Soc. Testing Mats., p. 65 (1953). (Issued as separate publication, *ASTM STP No. 155.*)

(3) "Electron Microstructure of Tempered Bainite and Tempered Martensite in Steel," Fourth Progress Report of Subcommittee XI of Committee E-4, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 568 (1954).

(4) D. M. Teague and S. T. Ross, "Electron Microstructures of Bainite and Tempered Martensite in Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 590 (1955).

## APPENDIX V

# THE TWO-STAGE PRESHADOWED CARBON POSITIVE REPLICA METHOD FOR ELECTRON METALLOGRAPHY*

By Ronald L. Scott[1] and Anna M. Turkalo[2]

## Synopsis

Techniques are described for obtaining high-resolution carbon replicas of a variety of surfaces ranging from very smooth to fairly rough contours. In addition to affording the advantages of good resolution and superior replica strength, the techniques need not destroy or mar the specimen surface. The replicating technique involves two stages: (1) the casting of a primary plastic negative replica, and (2) the evaporation upon the plastic replica of shadowing material followed by the evaporation of the carbon film. The plastic is finally dissolved, leaving the preshadowed carbon replica for viewing in the electron microscope. The relative merits of chromium and tungsten oxide as shadowing materials are discussed. Applications of the two-stage preshadowed carbon replica technique to ferrous and non-ferrous structures are shown.

The high resolution available with modern electron microscopes and the concurrent improvements in polishing and etching methods used to prepare specimens for electron metallography have placed an ever-increasing demand for better quality and resolution on the replica used. Consequently, a variety of replication methods have been developed to meet specific needs.

The primary requirement for successful electron metallography is faithful reproduction of the metal surface with a replicating technique which yields consistent results that can be unambiguously interpreted. The fulfilment of this objective depends to a large degree upon the following factors:

1. Intrinsic resolution of the replica;
2. Mechanical strength of the replica;
3. Ease of removal of the replica from the metal surface;
4. Stability of the replica in the electron beam;
5. Simplicity of the replicating method; and
6. Reproducibility of results.

Thus all these factors are important in the consideration of any new replication method.

The thin plastic negative replica, perhaps the most satisfactory of the methods evolved while electron metallography was in its early stages of development, is still popular with electron metallographers. This is a very simple, and in many cases quite adequate technique. However, there are two serious limitations to this type of replica: (1) The effective resolution afforded by the thin plastic material (50 to 100 Å) is considerably less than that allowed with the present-day electron microscope which can give as much as 15 to 20 Å resolution. (2) Thin plastic replicas suitable for viewing in the electron microscope cannot be stripped successfully and easily from many surfaces. The roughness of surface is the determining factor. A deeply etched or a rough fracture surface is difficult and often impossible to replicate with the thin plastic negative technique.

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* Presented at a meeting of Subcommittee XI on Electron Microstructure of Metals of ASTM Committee E-4 on Metallography, Atlantic City, N. J., June 18, 1957.

[1] Research Staff, General Motors Corp., Detroit, Mich.

[2] Research Laboratory, General Electric Co., Schenectady, N. Y.

The limitations of the plastic replica technique are overcome by the use of the recently introduced carbon replication techniques (**1**).[3] The carbon replica has superior resolution, high contrast, and exceptional mechanical strength both during preparation and during examination in the electron beam. In addition, it can be used in a two-step process to replicate a variety of surfaces without destruction of the specimen. Obviously, a replication technique possessing these features holds promise of meeting all needs of the electron metallographer and hence should be considered as a replacement for the older methods still in use.

Work with the two-step carbon replica in a number of new and different ways both at the research staff of the General Motors Corp. and at the research laboratory of the General Electric Co. has shown definite advantages over the direct-stripped, shadowed, plastic replica.

Recently Subcommittee XI, of ASTM Committee E-4 on Metallography, decided to reevaluate the relative merit of different replication techniques being used currently by members of the committee. For this purpose three structures in a plain carbon eutectoid steel (SAE 1087) were selected for replication by the various techniques. These were lower bainite, martensite tempered at 400 F, and a partial transformation structure of upper bainite and martensite. The authors were assigned the task of applying the two-step preshadowed carbon technique in the replication of the three structures. It is the purpose of this paper to report the results of this work as well as to show other applications of the technique in electron metallography.

## TECHNIQUE

In the two-step carbon method a plastic negative replica is made first. For this replica Parlodion, Faxfilm, or some other suitable plastic may be used. This replica is shadow-cast with an appropriate material and then carbon is evaporated on it normal to the surface. The plastic negative is subsequently dissolved away, leaving a preshadowed positive carbon replica.

### *Primary Replication:*

The preparation and handling of the primary plastic replica can be accomplished in a variety of ways. The following methods have been successfully used by the authors.

*Method 1.*—A 1 per cent Formvar solution in dioxane, or chloroform, is flowed over the specimen surface and is then allowed to dry with the specimen face positioned 90 deg to the horizontal. Then, over the first film, a second film is formed by using a 2 per cent solution of nitrocellulose in amyl acetate. The composite film is then dry-stripped with Scotch tape. Following this, the shadowing and carbon evaporations are performed. After carbon evaporation, the film is cut into $\frac{1}{8}$-in. squares and dropped into the nitrocellulose solvent to free the Scotch tape and second layer, leaving just the preshadowed carbon replica with the original Formvar film. The pieces are then caught on specimen grids, washed in the Formvar solvent by the Jaffe (2) method, leaving only the preshadowed carbon replica on the grid. In the Jaffe method the replicas are not completely immersed in the solvent; they are mounted on a stand in a closed vessel and washed from the bottom only. This procedure is less severe than complete immersion.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 553.

*Method 2.*—In this method a thin film of Parlodion is allowed to spread across the specimen surface. For this purpose a 1 per cent solution of Parlodion in amyl acetate is used. When this has dried completely, a few drops of 5 per cent Parlodion are placed on the specimen surface. The specimen is then tilted 45 deg from horizontal, allowing the excess solution to drain off. Before the second film is dry, 200-mesh screens are embedded in the resin. When the film is completely dry, the surface of the specimen is flooded with water and the films, with screens embededd, are teased free of the specimen surface. As an alternate method, dry-stripping can be used. It is sometimes feasible to make the original plastic replica directly with the 5 per cent solution. In this case, the specimen screen is embedded in this film. Dissolving of the replica after the positive carbon replica has been made is done by the Jaffe method.

*Method 3.*—This method uses Faxfilm applied in one of two ways. In the first, the Faxfilm is wet on one side with acetate and this side is pressed against the specimen with the thumb or a soft eraser for a few seconds until drying has begun. An alternate method is to completely immerse the Faxfilm in acetone and place it wet over the specimen surface and allow complete drying before stripping. After drying has occurred (1 to 10 min), the Faxfilm is easily lifted from the specimen surface, and the replica is ready for further processing.

The Faxfilm technique is especially well suited for relatively rough surfaces such as fractures from which one cannot strip the thinner films used in the first or second method. However, considerable difficulty may be encountered with rupturing of the carbon film when the Faxfilm technique is used. During the dissolving process the Faxfilm swells, breaking up the carbon film. It is interesting to note that this tendency is greatest in cases of smooth surfaces. A surface with a considerable number of irregularities appears to impart more flexibility to the carbon film. This resists stresses which are produced by the swelling of the dissolving Faxfilm. To inhibit the breaking up of the thin carbon film during the dissolution of the Faxfilm, the evaporation of a thin layer of a wetting agent such as Victawet is often beneficial. This is evaporated prior to the deposition of the preshadowing metal. The freeing of the preshadowed carbon replica from the Faxfilm is accomplished by cutting the Faxfilm-carbon composite into small squares which are placed on grids and washed with acetone by the Jaffe method. If the replica is oriented so that the carbon side is next to the grid and the plastic is facing outward, the carbon film will have less tendency to rupture during solution of the plastic.

In addition to the plastics just noted for the primary replica, methacrylate resin or other similar material may be used if it: (*a*) has no observable structure of its own, (*b*) replicates the surface topography faithfully, and (*c*) can be freed easily from the carbon replica.

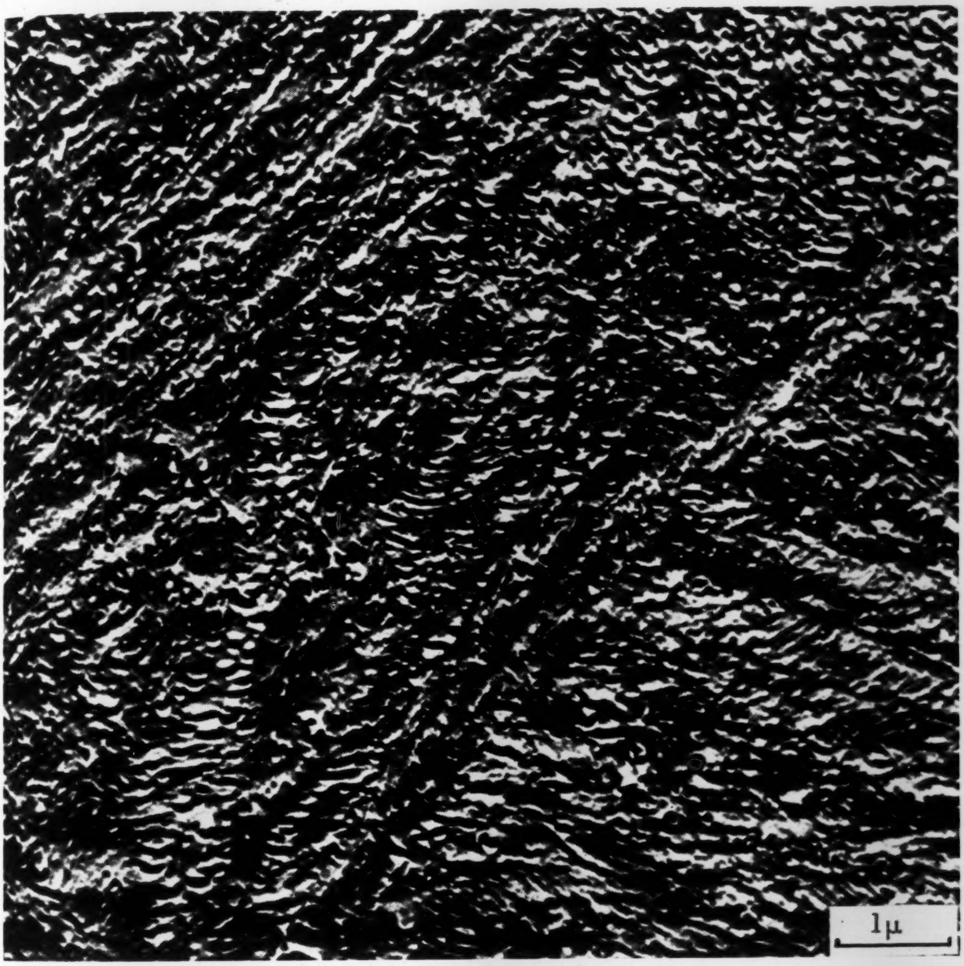

Fig. 1.—1087 Steel.

Bainite, isothermally transformed 2½ hr at 400 F;
Polish, mechanical and chemical;
Etch, 4 per cent picral;
Final replica, carbon;
Primary replica, Parlodion;
Shadowed with $WO_3$.

*Shadow-Casting:*

Shadowing to increase contrast and enhance resolution is accomplished by vacuum evaporation prior to the deposition of the carbon film. The vacuum is not broken between the deposition of the shadowing material and the evaporation of the carbon. For shadowing materials, either chromium or tungsten oxide is used. Chromium has been used as a shadowing material for some time and its performance is well known to electron metallographers. It is easily evaporated from a basket of 20-mil tungsten wire (3) or from a V-shaped tungsten wire, the apex of which has been electroplated with chromium. Tungsten oxide (**4,5**)

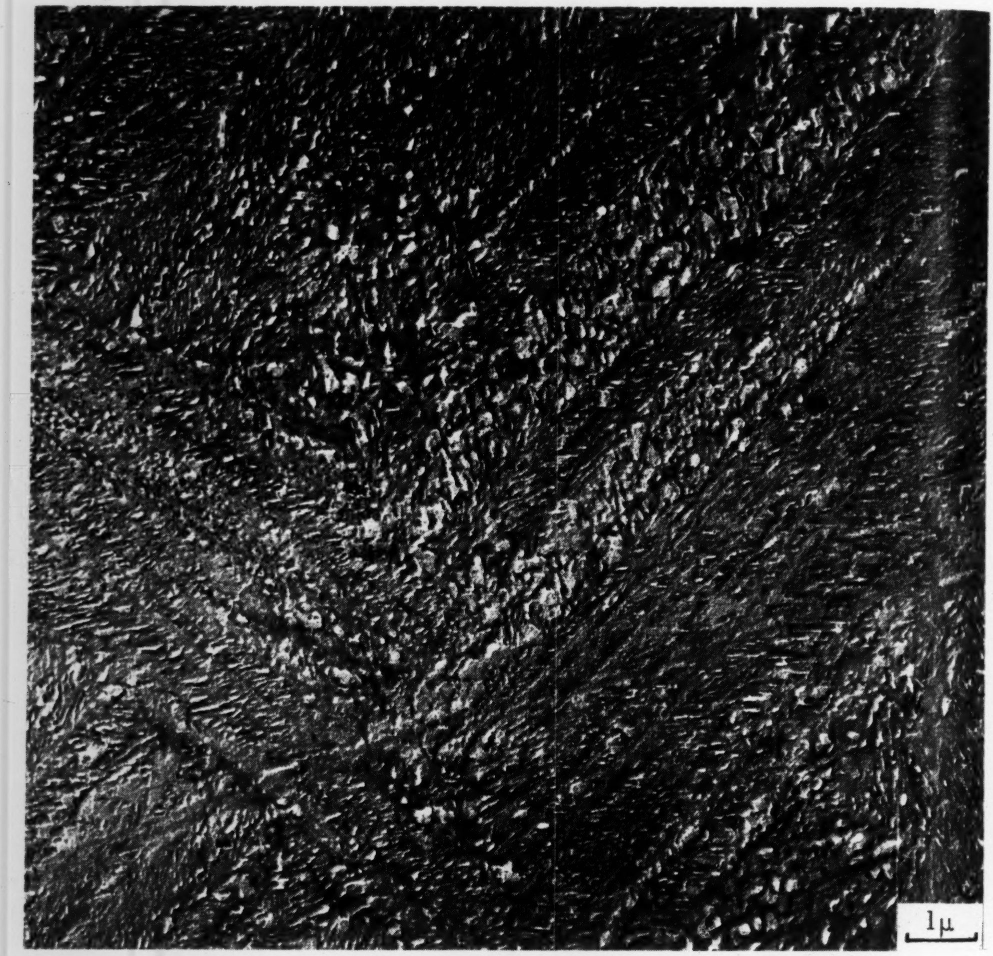

FIG. 2.—1087 Steel.

Bainite, isothermally transformed $2\frac{1}{2}$ hr at 500 F;
Polish, mechanical;
Etch, 4 per cent picral;
Replica, Formvar negative;
Shadowed with chromium.

has only recently been recommended as a shadowing material and therefore has not been used extensively. The filaments for shadowing this material are made from 10- or 20-mil tungsten wire bent to form a "V" in order to create, as nearly as possible, a point source. The filament is then placed in a vacuum and residual surface contaminants are burned off by passage of a current. Air is then admitted and the wire is again brought to red heat electrically, resulting in the formation of a citron-yellow deposit of tungsten trioxide on the wire. This is then evaporated in a manner similar to the evaporation of chromium.

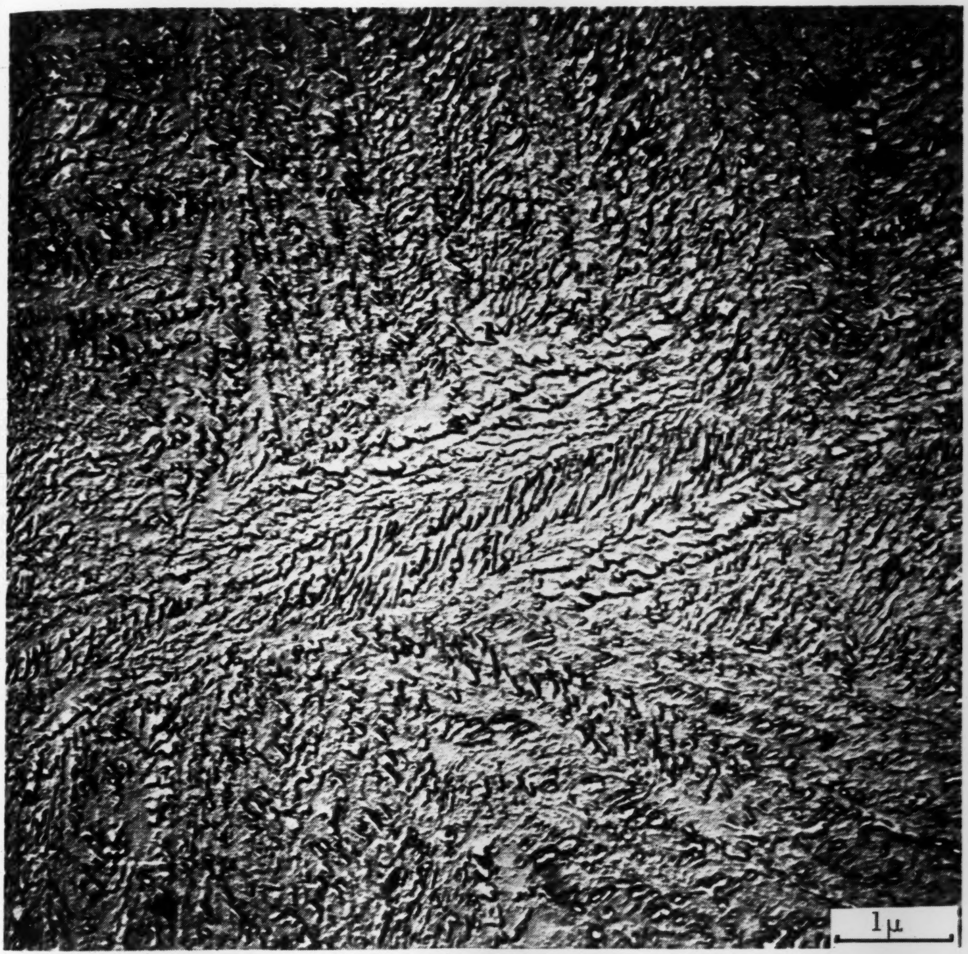

FIG. 3.—1087 Steel.

Bainite, isothermally transformed $2\frac{1}{2}$ hr at 500 F;
Polish, mechanical;
Etch, 2 per cent picral;
Final replica, carbon;
Primary replica, Formvar;
Shadowed with chromium.

A small amount of tungsten oxide gives replicas of very high contrast. In fact, replicas of deeply etched surfaces comprised of very fine structure have such high contrast that there is difficulty in interpretation (Fig. 1). Increasing the shadowing angle, with respect to the plane of the replica, results in a shorter shadow and helps to correct this condition. The most satisfactory tungsten oxide shadowing occurs when the filament-to-specimen distance does not exceed 2 in., particularly where high contrast is desired. The shadowing effect drops off rapidly beyond 3 in. The advantage of tungsten oxide, apart from

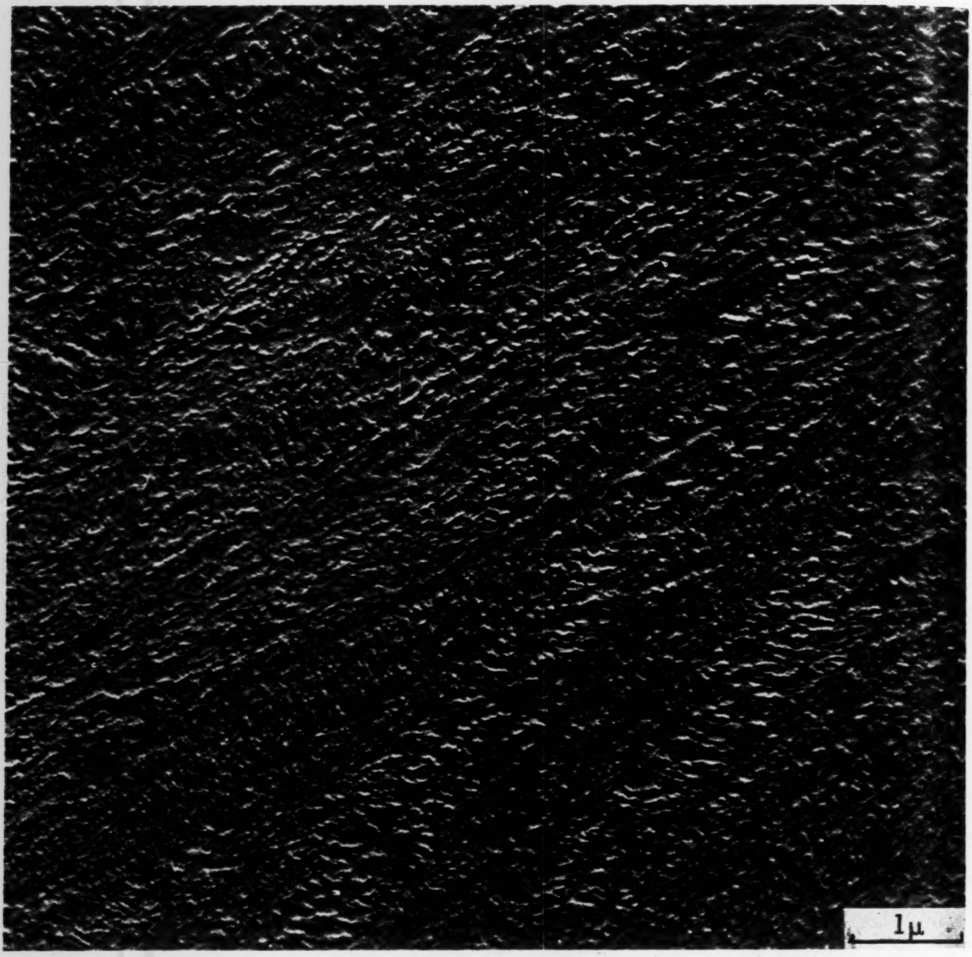

FIG. 4.—1087 Steel.

Bainite, isothermally transformed $2\frac{1}{2}$ hr at 500 F;
Polish, mechanical;
Etch, 4 per cent picral;
Final replica, carbon;
Primary replica, Parlodion;
Shadowed with $WO_3$.

the simplicity of filament preparation, lies in the wide range of contrasts available to the metallographer.

*Carbon Replication:*

The carbon replica obtained by evaporation was first introduced by Bradley in 1954. Its various uses are discussed by him in a more recent paper (6) which indicates the versatility of the process.

Early in our use of the two-stage carbon replica the results were often disappointing. It frequently happened that during removal of the plastic negative in the final step, the carbon replica was destroyed or severely damaged. In subsequent discussions with other in-

FIG. 5.—1087 Steel.

Martensite tempered 1 hr at 400 F;
Polish, mechanical;
Etch, 2 per cent picral;
Final replica, carbon;
Primary replica, Formvar;
Shadowed with chromium.

vestigators it was found that this was a common complaint. As a result, many metallographers were returning to the more conventional methods of replication.

It was first believed that this difficulty was associated either with the rate of deposition of the carbon or with carbon thickness. Control of these variables through regulation of the current was not possible using the method employing two pointed electrodes, as recommended by Bradley. By using one pointed electrode and one flat electrode, with spring loading to maintain electrode contact during evaporation, control over the evaporation current was possible. This enabled determination of

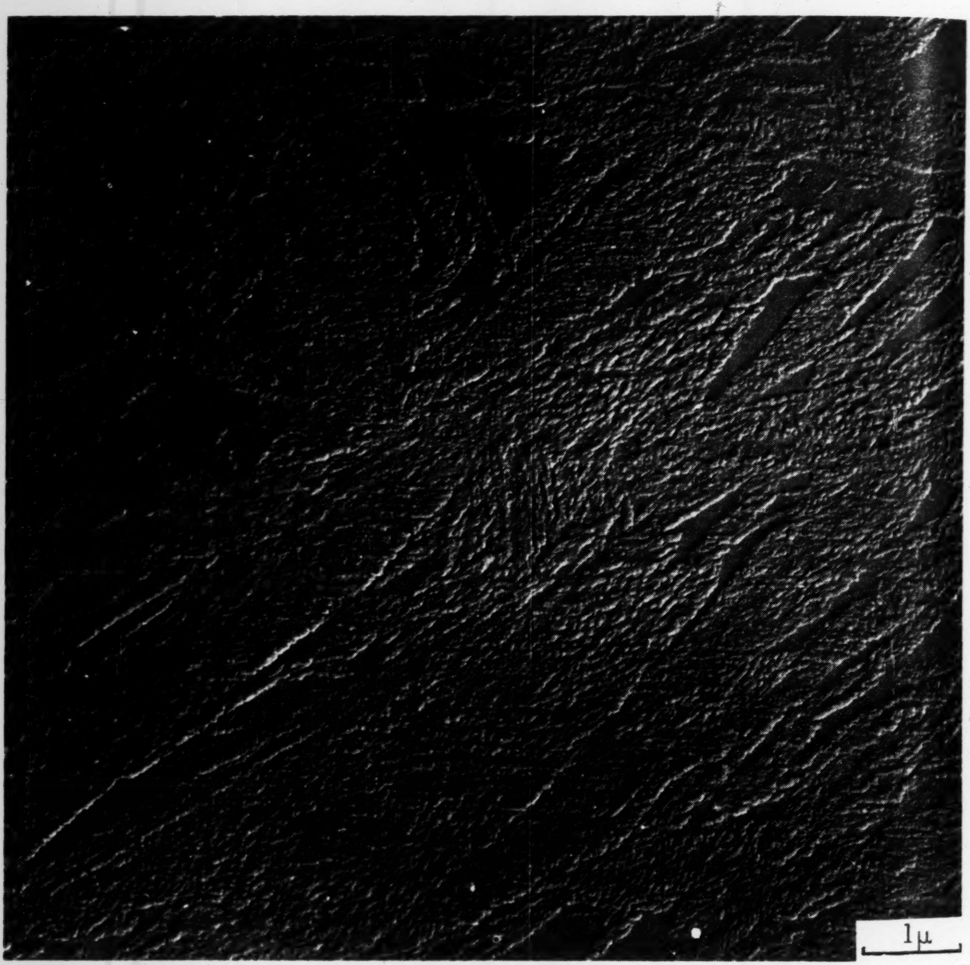

FIG. 6.—1087 Steel.

Martensite tempered 1 hr at 400 F;
Polish, mechanical;
Etch, super picral;
Final replica, carbon;
Primary Replica, Parlodion;
Shadowed with $WO_3$.

the optimum evaporation conditions for a given equipment geometry. Independent investigations using the two-stage carbon replica method show, however, that widely varying rates of evaporation and carbon films of various thicknesses produce comparable results. Using a distance of 4 in. from replica to evaporation point, currents of from 30 to 120 amp flowing for 5 to 20 sec result in high yields of good replicas. The color of the carbon films range from yellow for a thin film to a metallic luster for thick films (~500 Å).

The major factor contributing to the destruction of replicas in the final step is now thought to be the thickness of the plastic replica being dissolved. Solution

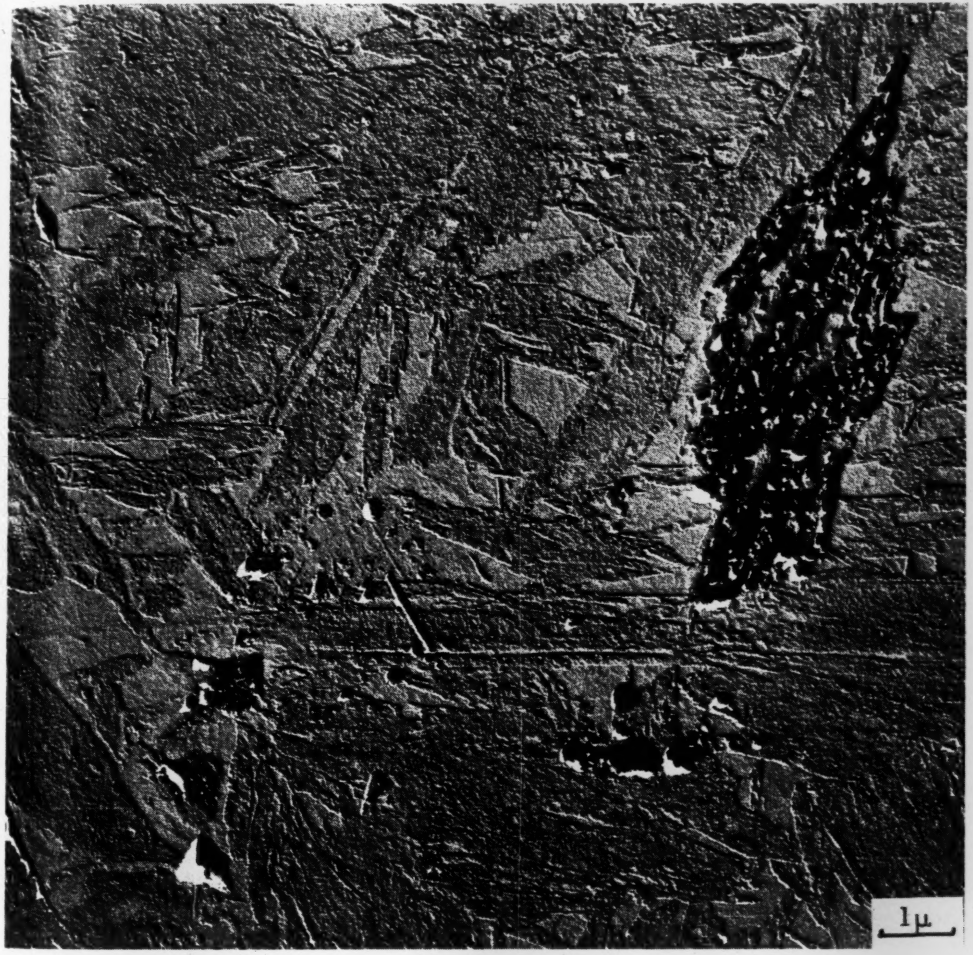

FIG. 7.—1087 Steel.
Bainite and martensite, isothermally transformed 7 min at 600 F;
Polish, mechanical;
Etch, heavy super picral;
Replica, Formvar negative.

of the plastic is accompanied by a swelling action resulting in stresses which, if sufficiently great, will tear the carbon apart. Experimental data indicate that a thin (yellow) film of carbon is strong enough to produce a good replica when the primary replica is made by method 1 (Formvar). Method 2, however, requires the heavier deposit of carbon exhibiting a metallic luster for consistently satisfactory replicas. Additional strength is imparted to the replica by embedding the replica support screen at the outset. This embedding technique is also useful for replication of a selected area of the specimen. Faxfilm, being a relatively thick material (about 5 mil), requires additional care if satisfactory results are to be achieved. The evaporation of a film of Victawet and the plac-

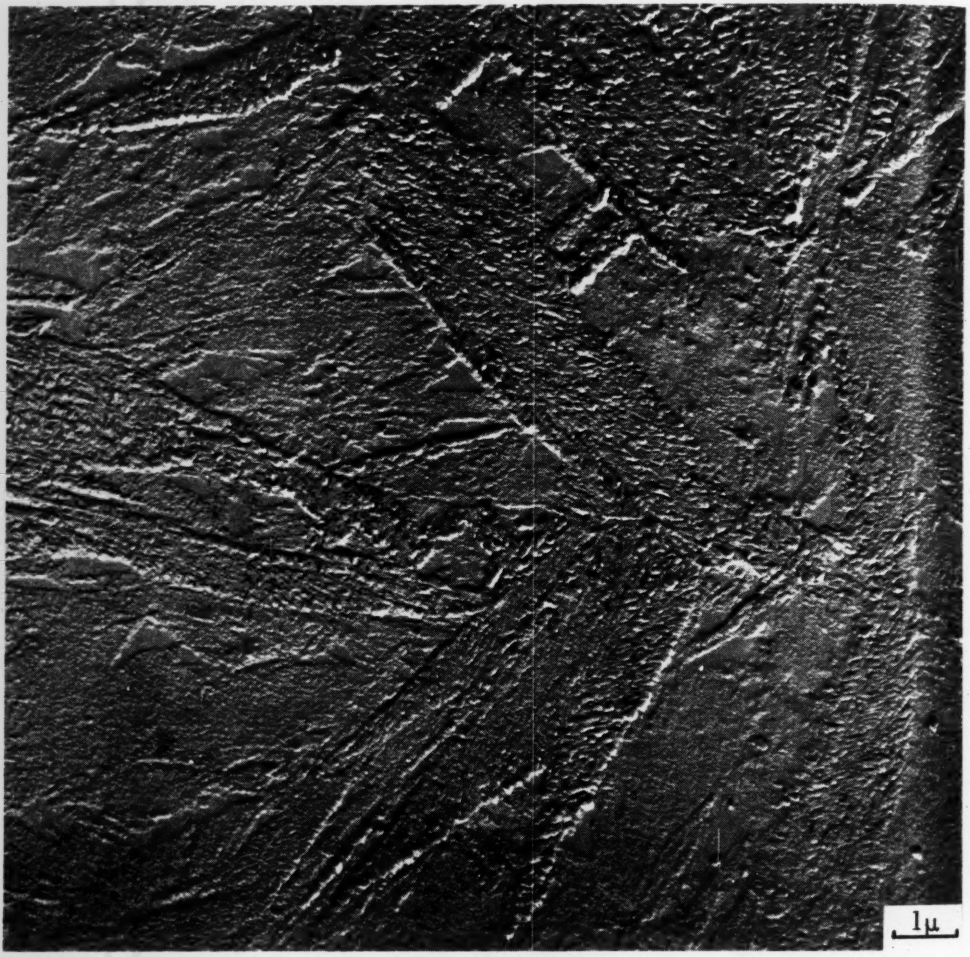

FIG. 8.—1087 Steel.

Bainite and martensite, isothermally transformed 12 sec at 850 F;
Polish, mechanical;
Etch, super picral;
Final replica, carbon;
Primary replica, Parlodion;
Shadowed with $WO_3$.

ing of the carbon side of the replica next to the support screen, as previously mentioned, increase the probability of successful replication.

## DISCUSSION

The procedures described produce carbon replicas of excellent quality. The techniques allow replication of deeply etched specimens having undercut or sharply reentrant surfaces, and result in high yields of good replicas free of strain marks and tears. Although the two-step method is not as simple as the single plastic negative method, the additional complexity is usually justified by the quality of the replicas obtained from surfaces which, in some instances, could

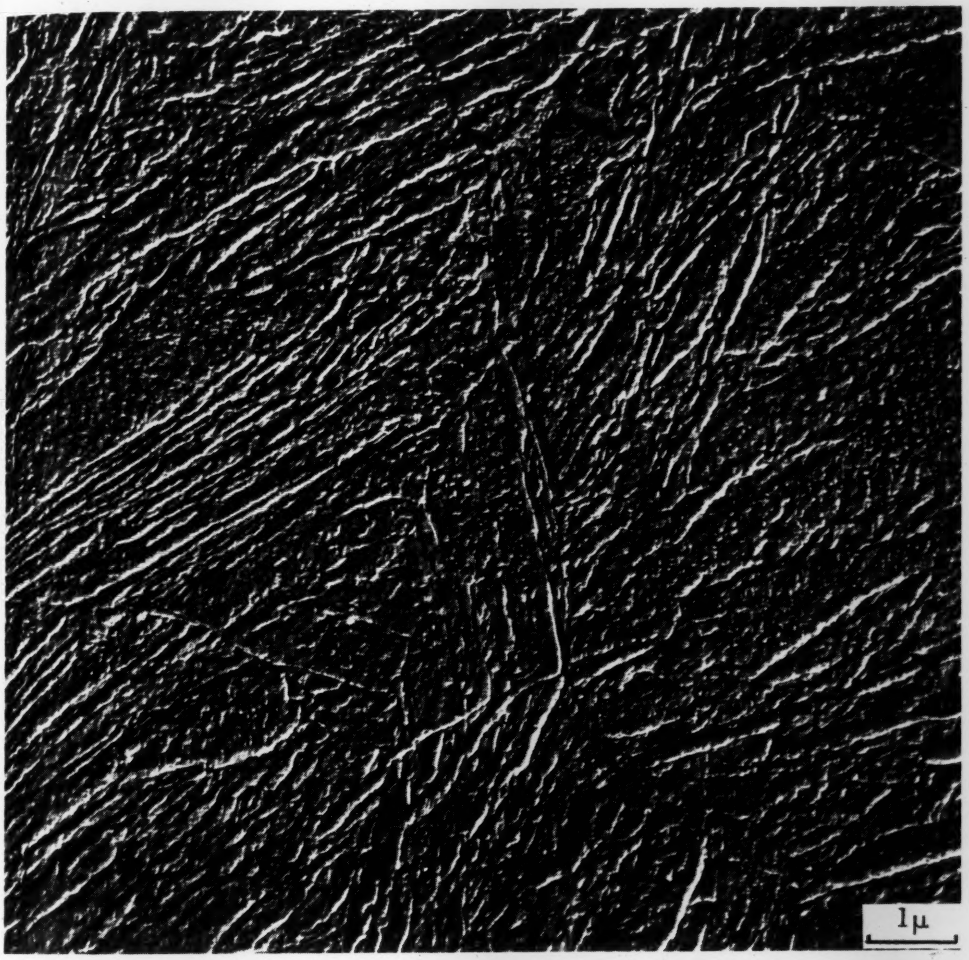

FIG. 9.—4140 Steel.

Martensite tempered 1 hr at 300 F;
Polish, electrolytic;
Etch, super picral;
Final replica, carbon;
Primary replica, Parlodion;
Shadowed with $WO_3$.

not be replicated by simpler methods. Stability of the carbon film in the electron beam is excellent. This permits replicas to be viewed for long periods of time at high beam intensity, with no apparent adverse effects on the replica. This stability as well as mechanical strength allows reexamination of the replica, if necessary. All of these properties culminate in an economical replicating procedure which gives consistent and reliable results that can be interpreted.

Figures 2 to 12 demonstrate some of the results obtained with the two-step preshadowed carbon technique used in replicating a number of microstructures.

Figures 2, 3, and 4 show the structure of lower bainite formed at 500 F in

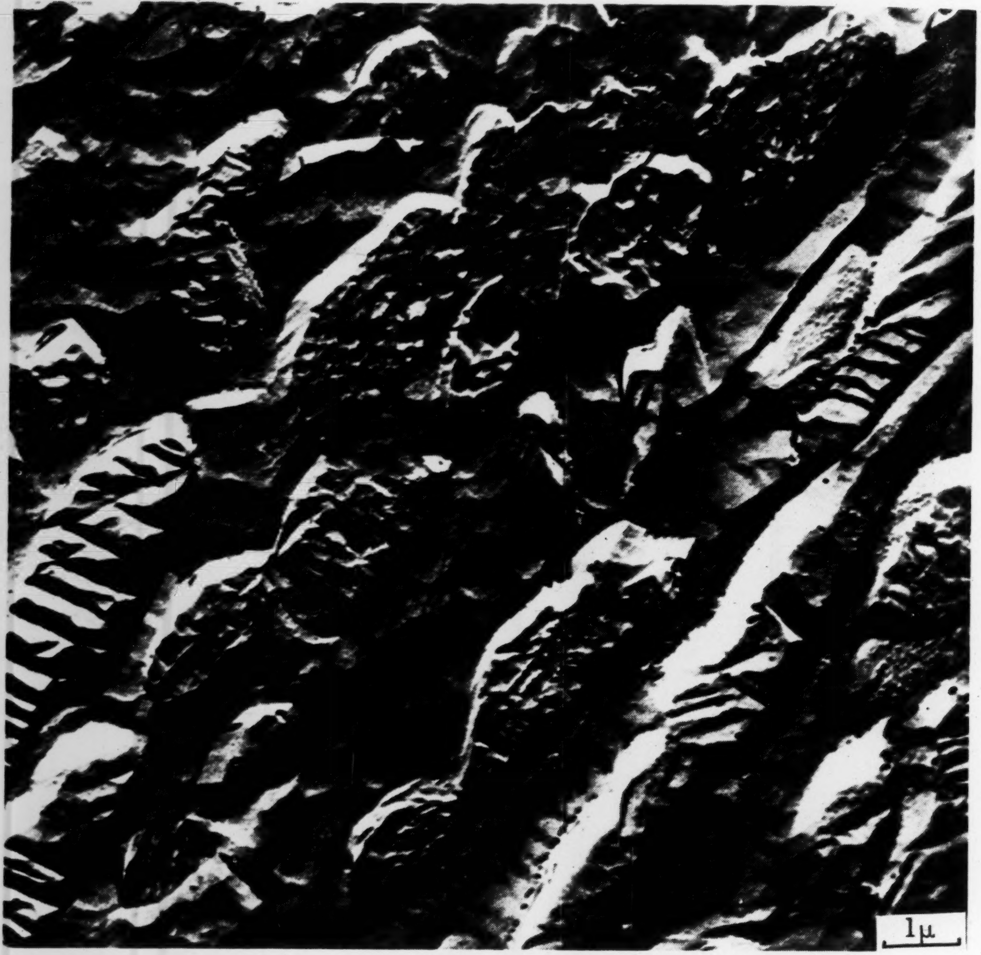

FIG. 10.—Titanium Alloy with 8 per cent Manganese.

Furnace cooled 5 deg per min to 1050 F;
Air cooled 1050 F to room temperature;
Polish, mechanical;
Etch, 2 per cent HF, 4 per cent $HNO_3$;
Final replica, carbon;
Primary replica, Parlodion;
Shadowed with $WO_3$.

SAE 1087 steel. This is one of the specimens selected by Subcommittee XI for replication. Figure 2, produced by the negative plastic replica technique, is included for comparison. Figure 3 illustrates results obtained using method 1 to produce the primary replica. Figure 4 is a micrograph of the same structure replicated by method 2 and shows that carbon replicates the microstructure faithfully even when a light etch is used.

Figures 5 and 6 are micrographs of martensite in a 1087 steel quenched and tempered at 400 F. This also is a structure recommended for replication by the committee. Two of the primary replica

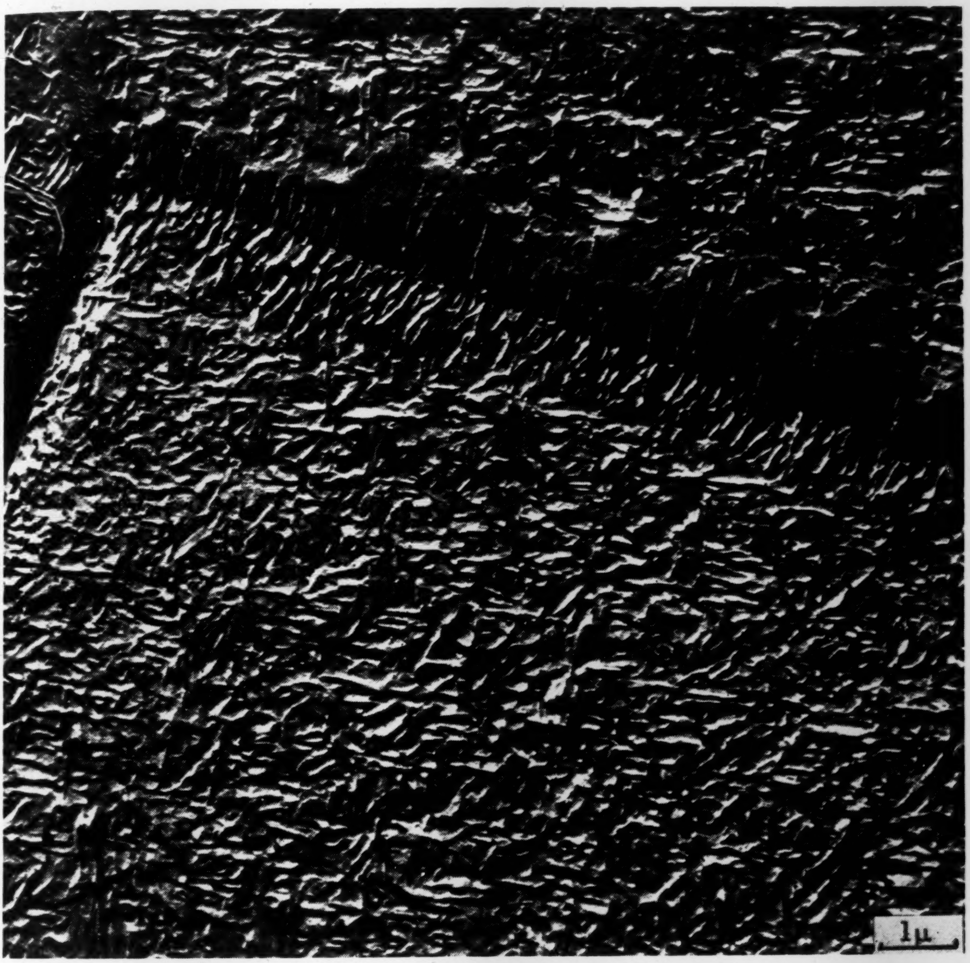

FIG. 11.—Titanium Alloy with 8 per cent Manganese.

Quenched from 1450 F;
Aged 2 hr at 1000 F;
Polish, mechanical;
Etch, 2 per cent HF, 4 per cent $HNO_3$;
Final replica, carbon;
Primary replica, Parlodion;
Shadowed with $WO_3$.

methods are represented as well as both shadowing materials. The two phases present are seen equally well, and the fine structure within the martensite grains is easily discernible.

Figures 7 and 8 are illustrations of specimens chosen by the committee for replication. Figure 7, made from a shadowed negative plastic replica, shows the etching difficulties encountered with mixed structures of bainite and martensite; that is, in order to show the structure of martensite and retained austenite it is usually necessary to overetch the bainite, obliterating the internal structure. Figure 8 demonstrates the high

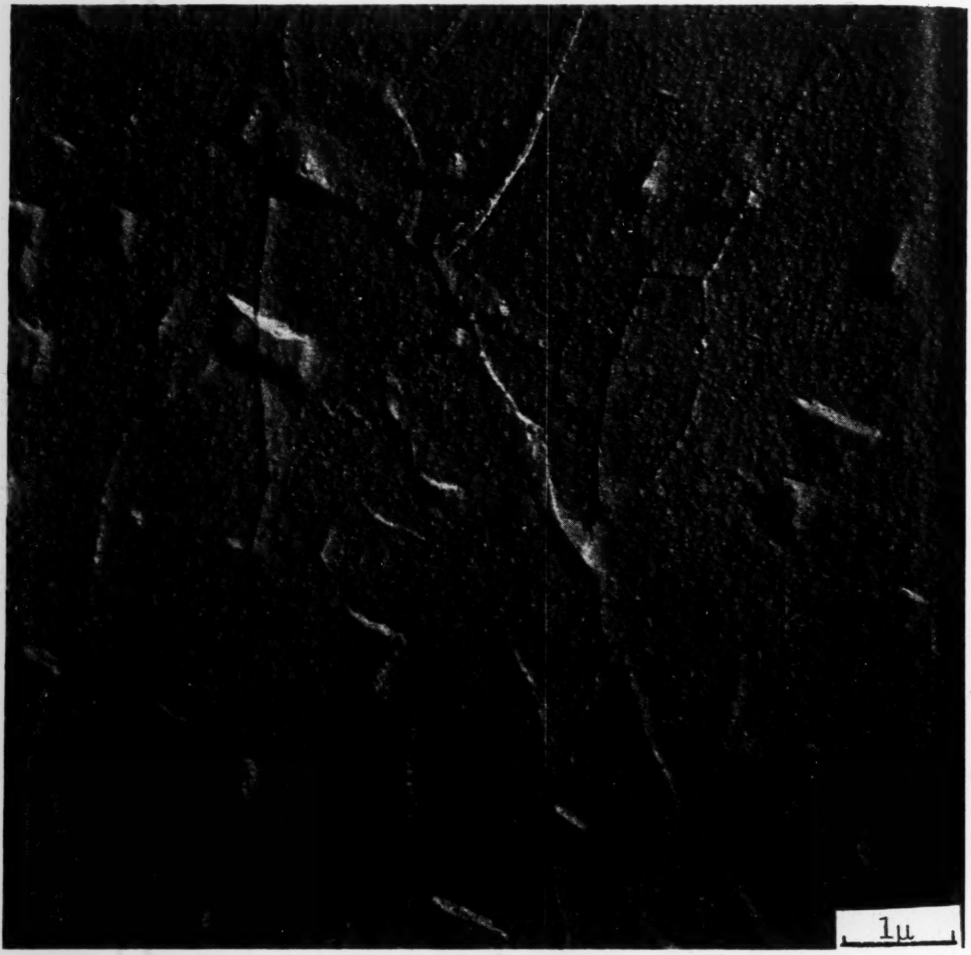

FIG. 12.—Low Carbon (0.03 per cent) Enameling Iron, Cold Reduced 10 per cent.
Polish, mechanical and electrolytic;
Etch, 1 per cent nital;
Final replica, carbon;
Primary replica, Parlodion;
Shadowed with chromium.

resolution of the two-stage carbon replica which enables observation of all three phases using a light etch.

Figure 9 shows the thin films of epsilon iron carbide and fine intragranular precipitate in quenched and tempered martensite of SAE 4140 steel.

Figures 10 and 11 are two micrographs of a manganese-titanium alloy. The heat treatments, radically different, have produced alpha particles of widely different sizes, both of which are easily replicated without tears and strain marks in the replicas. The high contrast available with tungsten oxide shadowing is illustrated in Fig. 10.

Figure 12 is a micrograph of low carbon (0.03 per cent) enameling iron showing sub-boundaries in the ferrite and nitride particles.

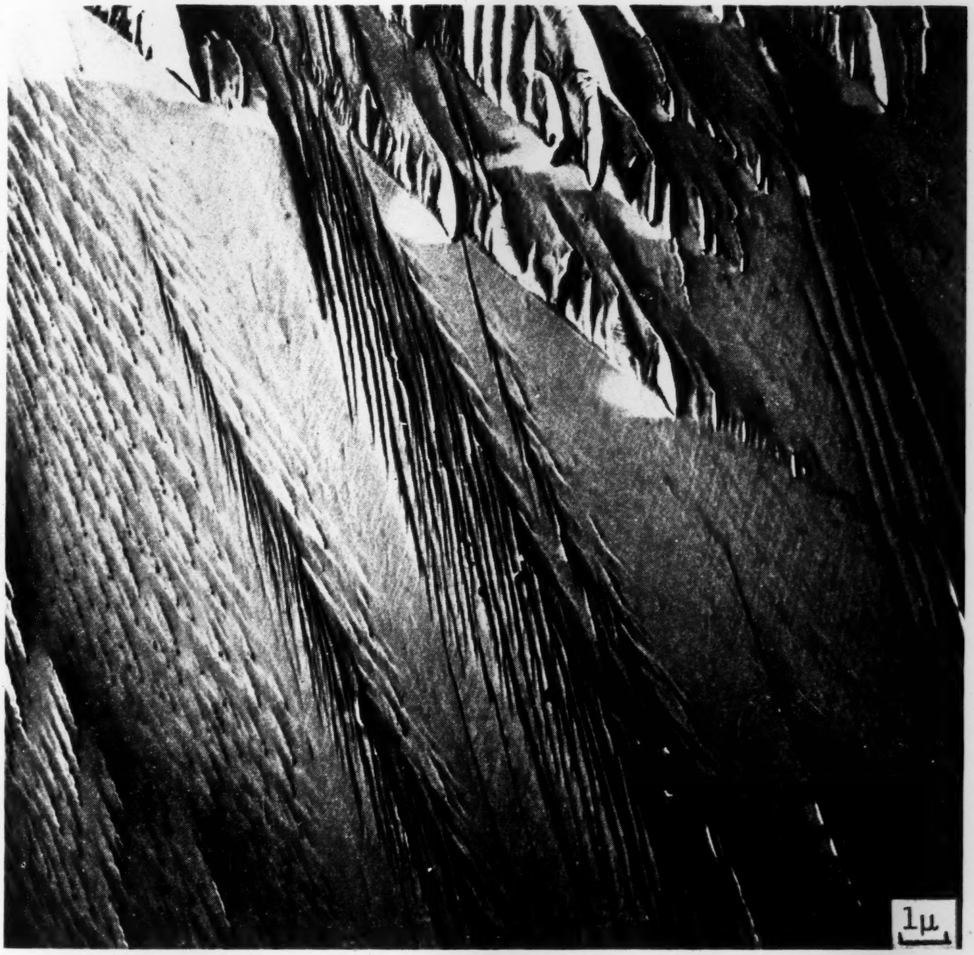

FIG. 13.—Cleavage Fracture Surface in 50 Nickel-50 Aluminum Alloy.
Final replica, carbon;
Primary replica, Faxfilm;
Shadowed with chromium.

Figures 13 and 14 show two micrographs of fracture surfaces of a nickel-aluminum alloy. Figure 13 is a cleavage portion and Fig. 14 is an illustration of intergranular fracture showing a grain boundary phase. Faxfilm was used as the primary replica.

## CONCLUSIONS

From our experience with the two-stage preshadowed carbon positive replica the following conclusions can be made:

1. The techniques described here make it possible to obtain carbon replicas of a variety of different surfaces having a wide range of contour without marring the surface during preparation.

2. Replicas made by the method described possess good mechanical strength and excellent resolution. In addition,

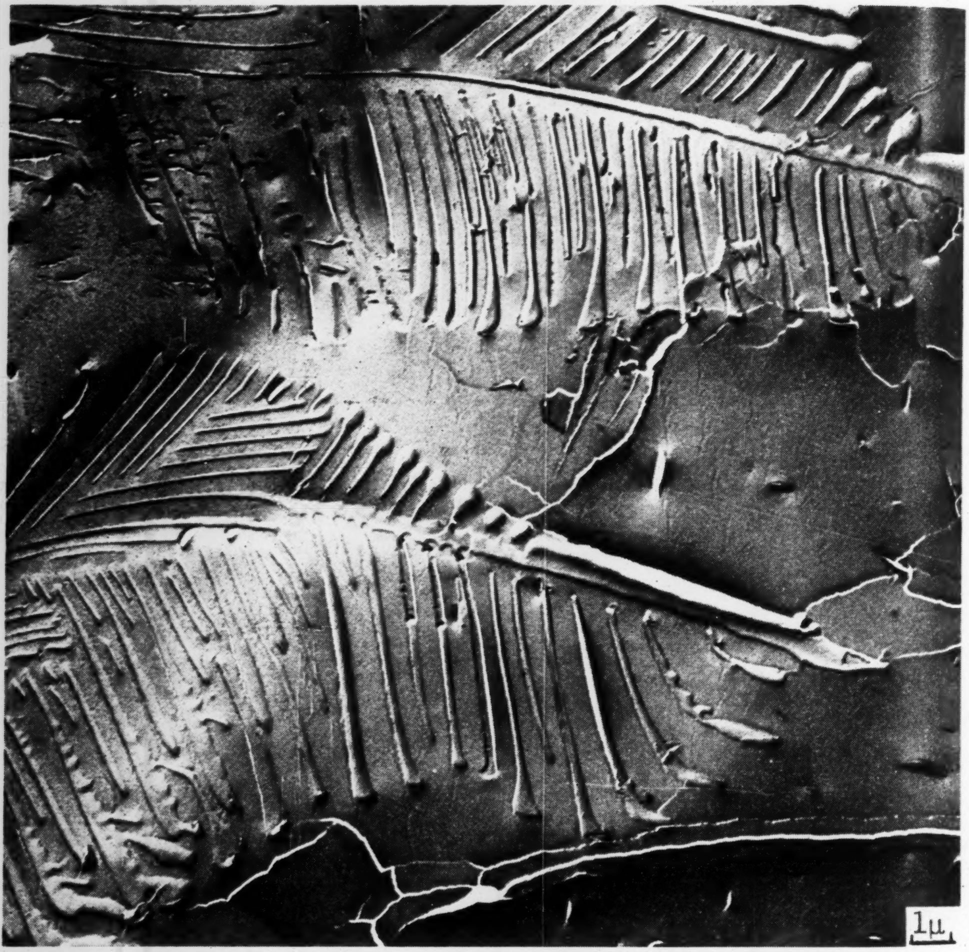

FIG. 14.—Intergranular Fracture in 50 Nickel-50 Aluminum Alloy.

Final replica, carbon;
Primary replica, Faxfilm;
Shadowed with chromium.

they exhibit an ability to resist breakdown in the electron beam not possessed by plastic replicas.

3. By exercising nominal care in preparation of the primary replica and control of carbon deposition, consistently high yields of excellent replicas are obtained with this method.

4. As a shadowing material, tungsten oxide gives a greater contrast range than chromium. In addition, the evaporation filaments are easily prepared.

## REFERENCES

(1) D. E. Bradley, "A High-Resolution Evaporated-Carbon Replica Technique for the Electron Microscope," *Journal*, Inst. Metals, Vol. 83, p. 35 (1954–1955).

(2) Mary S. Jaffe, "Proceedings of Electron Microscope Society of American Meeting," *Journal of Applied Physics*, Vol. 19, p. 1191 (1948).

(3) R. C. Williams and R. W. G. Wyckoff, *Science*, Vol. 101, pp. 594–596 (1945).

(4) F. Van Wijk, "Metallographie Electronique du Fer Pur et de l'Acier Doux," *Revue Universelle des Mines*, Series 9, Vol. XII, p. 555, Oct., 1956.

(5) H. Wilsdorf, *Zeitschrift Metallkunde*, Vol. 45, p. 14 (1954).

(6) D. E. Bradley, "Uses of Carbon Replicas in Electron Microscopy," *Journal of Applied Physics*, Vol. 27, No. 12, pp. 1399–1412, Dec., 1956.

# REPORT OF COMMITTEE E-6

ON

# METHODS OF TESTING BUILDING CONSTRUCTIONS*

Committee E-6 on Methods of Testing Building Constructions held one meeting during the year: on February 7, 1957, in Philadelphia, Pa., in conjunction with ASTM Committee Week.

The committee has been going through a period of reorganization and has just completed a reconstitution of subcommittees, reconfirmation of membership interest, and a revision of scope. The revised scope reads as follows:

> *Scope.*—To formulate methods of test for building (including housing) construction, including elements, connections and assemblies, under actual or simulated service conditions, applicable to the evaluation of such factors as materials, design, construction and fabrication with special reference to the needs of building code and similar authorities with whom the committee has liaison.

Because of the expressed need of building officials concerned with new building codes, products and conditions, the anticipated activities of Committee E-6 will more closely conform to (and provide liaison with) the activities of the important building code groups of this continent.

Without conflict of interest with any established ASTM committee, Committee E-6 has established liaison with such as are interested in building constructions so that, as necessary when problems arise, an *ad hoc* committee may be mutually manned to seek answers. Eight subcommittees are now active or about to be activated:

I. Subcommittee on Panels for Lightweight Building Construction (J. A. Liska, Chairman).
II. Subcommittee on Durability (E. C. Shuman, Chairman).
III. Subcommittee on Sound Transmission (R. K. Cook, Chairman).
IV. Subcommittee on Masonry (H. C. Plummer, Chairman).
V. Subcommittee on Trusses (D. E. Kennedy, Chairman).
VI. Subcommittee on Load Tests on Full Scale Trusses (W. R. Schrieves, Chairman).
VII. Subcommittee on Vapor Barrier Under Slabs (R. F. Luxford, Chairman).
VIII. Subcommittee on Windows (R. B. Crepps, Chairman).

In September of 1956, a successful symposium covering Full Scale Tests on House Structures was held at the Los Angeles Meeting, jointly with Committee D-7 on Wood. It is expected the papers presented will be published as a booklet covering these proceedings.[1]

In 1958 at the Annual ASTM Meeting in Boston a Symposium on Durability is proposed.

In October of 1957, participation in a joint meeting has been planned with

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Symposium on Full-Scale Tests on House Structures, Am. Soc. Testing Mats. (1956). (Issued as separate publication *ASTM STP No. 210.*)

Committees C-16 on Thermal Insulating Materials and C-20 on Acoustical Materials at the National Research Council of Canada in Ottawa.

This report has been submitted to letter ballot of the committee, which consists of 82 voting members; 50 members returned ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. F. LEGGET,
*Chairman.*

R. A. BIGGS,
*Secretary.*

# REPORT OF COMMITTEE E-9
ON
# FATIGUE*

Committee E-9 on Fatigue held two meetings during the year on June 20, 1956 in Atlantic City, N. J., and on February 4, 1957 in Philadelphia, Pa.

Mr. W. S. Hyler of Battelle Memorial Inst. has been elected to membership. The committee consists of 50 members.

*Subcommittee I on Research* (T. J. Dolan, chairman) sponsored a talk before Committee E-9 by Mr. Egon Orowan on "The Physical Theory of Fatigue." Additional presentations along this line will be scheduled.

*Subcommittee II on Papers* (F. M. Howell, chairman).—The following papers relative to fatigue are being presented at the 1957 Annual Meeting. The first four papers are included in a general session on Fatigue, and the others constitute a Symposium on Large Fatigue Testing Machines and Their Results.[1]

"Cyclic Strain Fatigue Studies on AISI Type 347 Stainless Steel," by E. E. Baldwin, G. Sokol, and L. G. Coffin, Jr.

"The Fatigue Properties of Decarburized Steel," by G. T. Horne and H. A. Lipsitt.

"Cracking of Notch Fatigue Specimens," by M. S. Hunter and W. G. Fricke.

"Effect of Grinding Conditions and Resultant Residual Stresses on the Fatigue Strength of Hardened Steel," by L. P. Tarasov, W. S. Hyler, and H. R. Letner.

"Fretting Fatigue Strength of Titanium Alloy RC 130 B," by H. W. Liu, H. T. Corten, and G. M. Sinclair.

"A Unique Machine for Large Scale Fatigue Testing," by H. V. Cordiano.

"Torsional Fatigue Testing of Axle Shafts," by E. J. Eckert.

"Fatigue Testing of Airframe Structural Components," by H. W. Foster.

"Fatigue Performance of Marine Shafting Laboratory and Service Tests," by T. W. Bunyan.

"Fretting Corrosion of Large Shafts as Influenced by Surface Treatments," by O. J. Horger and H. R. Neifert.

"Fatigue Tests of Large Alloy Steel Shafts," by F. C. Eaton.

"A Quarter Century of Propulsion Shafting Design Practice and Operating Experience in the U. S. Navy," by R. Michel.

*Subcommittee III on Survey* (H. J. Grover, chairman).—During the year the 1955 References on Fatigue were published as ASTM STP No. 9-G. Consideration is being given to the preparation of a cumulative index of the material available.

*Subcommittee IV on Large Machines and Test Correlation* (J. M. Lessells, chairman) arranged for the Symposium on Large Fatigue Testing Machines and Their Results referred to under Subcommittee II.

*Subcommittee V on Aircraft Structures* (P. Kuhn, chairman).—Consideration is being given to publishing a pamphlet containing the papers on fatigue problems in the aircraft field as presented at Los Angeles, September 22, 1956.[2]

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The papers presented at the session on Fatigue appear in this 1957 *Proceedings*; the symposium papers are issued as Special Technical Publication, *ASTM STP No. 216* entitled Symposium on Large Fatigue Testing Machines.

[2] Second Pacific Area Meeting Papers on Fatigue of Aircraft Structures, 1956. (Issued as Special Technical Publication, *ASTM STP No. 203*.)

*Task Group on Statistical Methods* (F. B. Stulen, chairman) has prepared a Guide for Fatigue Testing and the Statistical Analysis of Fatigue Data.

This report has been submitted to letter ballot of the committee, which consists of 50 members; 35 members returned their ballots, of whom 35 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
*Chairman.*

O. J. HORGER,
*Secretary.*

# REPORT OF COMMITTEE E-12

ON

# APPEARANCE*

Committee E-12 on Appearance held two meetings during the year: in Atlantic City, N. J. on June 18, 1956, and in New York, N. Y. on March 5, 1957.

During the year, Committee E-12 has been concerned primarily with methods of color measurement, the relation of appearance specifications to consumer interest, and the preparation of an ASTM Manual on Appearance Properties of Materials. The results of a questionnaire circulated to all technical committees of the Society indicate considerable interest in the preparation of a Manual, and a meeting will be held on June 17, 1957, at the Annual Meeting of the Society at which representatives from the committees will be asked to discuss their needs for such a manual.

The services of Committee E-12 to aid in the solution of appearance problems were offered to committees of the Society and to industry. As a result, Committee E-12 has expanded its activities to cover a project concerning instrumental grading of the color of coal-tar resins.

This report has been submitted to letter ballot of the committee, which consists of 79 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

M. Rea Paul,
*Chairman.*

Richard S. Hunter,
*Secretary.*

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

# REPORT OF COMMITTEE E-13

## ON

# ABSORPTION SPECTROSCOPY*

Committee E-13 on Absorption Spectroscopy and subcommittees met in Pittsburgh, Pa., on March 4, 1957. The Advisory Committee met the previous evening to outline the future program of Committee E-13 and plans to meet again in New York during September, 1957.

Maintenance of the ASTM Punched-Card Index of Absorption Spectral Data is still the outstanding activity of the committee. The card-index system was developed by L. E. Kuentzel (now vice-chairman of the committee) of Wyandotte Chemicals Corp. The system is designed for IBM machine sorting of spectral absorption data to match spectrograms in qualitative chemical identification and chemical structure correlation. Gathering and coding of spectral data from the literature and from readily available institutional and commercial files is the responsibility of Subcommittee III on Standard Data (M. V. Otis, chairman). Card punching and distribution are administered by Society Headquarters. The complete index, now comprising over 35,000 cards, covers the ultraviolet and infrared ranges of absorption spectra and includes a Formula-Name Index to the infrared cards. The subcommittee plans to include visible, and perhaps near-infrared, spectral data in the index.

### New Tentative

The committee recommends for publication as tentative the Definitions of Terms and Symbols Relating to Absorption Spectroscopy as appended hereto.[1]

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

### Subcommittee Activities

A summary of the activities of subcommittees will appear in the ASTM Bulletin. In addition to the work on the index to spectral data under Subcommittee III, other subcommittees have been active in formulating nomenclature and practices in absorption spectrometry.

*Subcommittee I on Apparatus Specifications* (G. M. Wyman, chairman) has prepared recommended test methods for spectrophotometers which will be published as information following approval by the main Committee. The methods cover (1) stray radiation, (2) spectral resolution, (3) wavelength accuracy, (4) wavelength reproducibility, and (5) photometric accuracy and reproducibility. Task groups are developing test methods for absorption cells and for spectrofluorometers.

*Subcommittee II on Methods* (R. T. O'Connor, chairman) has prepared general techniques of infrared quantitative analysis and a suggested format for ASTM methods involving absorption spectrophotometry; both are subject to subcommittee approval. Work is under way on schemes of ultraviolet quantitative analysis.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 5.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

*Subcommittee IV on Nomenclature* (W. P. Tyler, chairman) prepared the proposed Tentative Definitions of Terms and Symbols Relating to Absorption Spectroscopy referred to earlier in the report. Additional definitions are being considered.

This report has been submitted to letter ballot of the committee, which consists of 65 members; 64 members returned their ballots, of whom 60 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

E. J. ROSENBAUM,
*Chairman.*

R. F. ROBEY,
*Secretary.*

# REPORT OF COMMITTEE F-1

ON

# MATERIALS FOR ELECTRON TUBES AND SEMICONDUCTOR DEVICES*

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices held three meetings during the year: in Cleveland, Ohio, on October 4 and 5, 1956; in Washington, D. C., on January 31 and February 1, 1957, and in Boston, Mass., on June 6 and 7, 1957.

At the Washington meeting a most interesting talk was given by J. O. McNally of the Bell Telephone Laboratories, Inc., on "An Electron Tube for a Repeatered Submarine Telephone Cable System."

The committee consists of 65 members, of whom 30 are classified as producers, 24 as consumers, and 11 as general interest members.

C. L. Guettel was appointed chairman of Subcommittee VIII, Editorial, replacing G. S. Szekely who resigned this position.

Because of the increased scope of the work in Section C on Analyses of Subcommittee I on Cathode Materials, a new Subcommittee IX on Materials Analyses was organized with R. S. Kelly, chairman. Sections have been set up on wet chemical analysis, spectrochemical analysis, and gas analysis. Adequate liaison with other methods committees will be maintained and encouraged.

On recommendation of the committee, the Society will publish this year a compilation of standards and tentatives on electron tube and semiconductor materials under jurisdiction of the committee, together with other standards relating to these materials.

Liaison activities have been maintained with other committees of the Society, including Committees E-2 on Emission Spectroscopy, E-3 on Chemical Analysis of Metals, and D-9 on Electrical Insulating Materials.

## RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1956 Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards revisions of the Tentative Method of Test for Interface Impedance Characteristics of Vacuum Tube Cathodes (F 300 - 55 T). This revised method was accepted by the administrative committee on March 6, 1957.

## NEW TENTATIVES

The committee recommends for publication as tentative the following specifications as appended hereto:[1]

*Specifications for:*

Clear Nickel-Clad and Nickel-Plated Steel Strip for Electron Tubes,
Aluminum-Clad Steel Strip and Nickel-Steel-Aluminum Composite Strip for Electron Tubes,
Clear Nickel Strip for Electron Tubes, and
Carbonized Nickel Strip and Carbonized Nickel-Plated and Nickel-Clad Steel Strip for Electron Tubes.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] The new and revised tentatives appear in the 1957 Supplement to Book of ASTM Standards, Part 6.

## REVISIONS OF TENTATIVES

The committee recommends revisions as appended hereto[1] of the Specifications for Circular Cross-Section Nickel Cathode Sleeves for Electronic Devices (F 239 - 49 T)[2] and continuation of the specifications as tentative.

## TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following specifications since revisions are in process:

*Tentative Specifications for:*

Tungsten Wire Under 20 Mils in Diameter (F 288 - 54 T), and

Round Wire for Use as Electron Tube Grid Laterals and Verticals (F 290 - 54 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

## ACTIVITIES OF SUBCOMMITTEES

*Subcommittee I on Cathode Materials* (A. M. Bounds, chairman) prepared the revision of the chemical tables in Tentative Specifications F 239 - 49 T, referred to earlier in the report.

Attention has been directed to the need for developing a standard for adhesion between coating and base metal after processing.

*The Electrical Test Section* Task Group on Interface Impedance has developed definitions for interface impedance, resistance, and capacitance which have been submitted to Subcommittee VIII for editorial review.

This section is also actively concerned with a proposed design for a standard planar diode to establish a recommended practice for the evaluation of tube parts in such a structure. A group of such diodes was distributed to the National Bureau of Standards, and the Westinghouse and Philco companies for the development of test methods and determination of electrical characteristics.

Work continues on a proposed standard triode specification. A task group has been formed, and parts have been made available so that triodes may be tested by a number of participating companies.

*The Physical and Mechanical Test Section* of this subcommittee is now concerned with satisfactorily completing the specification for the disk cathode measuring device and techniques. The section will also consider physical properties of sleeves and will attempt to set numerical standards for certain critical properties.

The *Analyses Section* of this subcommittee was reorganized as Subcommittee IX on Materials Analyses as indicated earlier in this report.

During the year the Task Group on Gas Analysis issued drafts on the procedures for the determination of carbon, hydrogen, nitrogen, and oxygen in electronic nickel.

The Task Group on Wet Analysis has been concerned with the improved method for magnesium determination and will continue to evaluate the method until it is ready for section ballot. The determination of sulfur in cathode-nickel alloys and development of analytical procedures for the determination of minor constituents in phosphors are being undertaken by this group.

The Spectrographic Task Group is working on the analysis of tungsten in nickel, with the latest drafts of the procedures for spectrographic analyses being currently reviewed in Committee E-2, with the hope they will soon be ready for submission to letter ballot.

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[2] 1955 Book of ASTM Standards, Part 6.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This group will renew its activity with the establishment of a third standard nickel oxide sample by the National Bureau of Standards. Work on four secondary standards submitted by International Nickel Co. will be started.

In the *Data Section*, a statistical method of evaluating factory prove-in data of new melts in cathode material has been submitted. Reports on factory prove-in tests on current heats of 220 alloy have been reviewed. A request has been made to the International Nickel Co. to melt one or more heats of alloy 220 grade containing from 0.5 to 0.7 per cent cobalt, the purpose of this request being to correlate the effect of cobalt on the strength of cathodes.

The section is analyzing data on the vacuum-annealed cathode nickel and the hydrogen-annealed vacuum-melted nickel, to determine the superior method.

*Subcommittee II on Insulators* (C. M. Harman, chairman) is primarily concerned with studies of mica, making evaluations on both natural and synthetic types. Five companies have submitted reports on tests performed.

Specifications for alundum are currently being reviewed particularly with regard to particle size measurements.

A method for high-frequency testing of ceramics has been briefly discussed.

*Subcommittee III on Strip* (C. W. Horsting, chairman) prepared the four proposed tentative specifications for strip referred to earlier in this report.

The subcommittee is evaluating data received relative to the carbon adherence tests and is continuing work toward improving the test for measurement of gas content in strip materials.

*Subcommittee IV on Wire* (D. R. Kerstetter, chairman) continues its work to revise the tentative specifications on tungsten and other grid wires.

Several methods for determining "out-of-roundness" of fine wire are being evaluated but no conclusions have as yet been reached as to the relative merit of the methods.

*Subcommittee V on Metallic to Non-Metallic Seals* (V. J. DeSantis, chairman) has appointed a task group to develop a standard seal for testing purposes to provide data on stress, strength, and vacuum-tightness. In conjunction with this work, the definitions for "hermetic" and "vacuum" seals are expected to be standardized.

The subcommittee is currently drafting a complete specification for dumet wire including requirements for color, chemical composition, dimensions, expansion, physical properties, etc.

Round-robin tests have been conducted and several drafts reviewed for a proposed tentative specification for iron-nickel-cobalt alloys for glass sealing applications.

Preliminary discussions toward writing specifications for sealing glasses indicate the desirability of cooperating with glass manufacturers and others on Committee C-14 on Glass and Glass Products.

*Subcommittee VI on Semiconductors* (H. C. Theuerer, chairman) is investigating crystal growing techniques and furnace design for producing standard silicon test specimens. Test methods and specifications for germanium dioxide and germanium metal are also under study. Round-robin tests by the cooperating companies will include samples of low- and high-resistivity materials with a standard resistivity test now under consideration.

*Subcommittee VII on Luminescent Materials* (J. G. Koosman, chairman) is making progress in development of a test for wet adhesion. It is expected that a paper entitled "A Proposed Method of Measuring the Wet Adhesion of Monochrome Television Phosphor

Screens" will be submitted for publication as information in the ASTM BULLETIN. Tests for dry adhesion are also being investigated.

Particle size determination continues to be of interest, with three methods being evaluated. Other tests being considered are for bulk density and for body-color of P-4 phosphors.

*Subcommittee VIII, Editorial* (C. L. Guettel, chairman) has processed eleven methods and specifications for other subcommittees. It has made detailed reviews of the galley proofs of F 128 (Methods of Test for Sleeves and Tubing for Radio Tube Cathodes) and F 205 (Methods of Test for Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps). A preliminary review of the proposed Method of Measurement of Wet Adhesion of Monochrome Television Phosphor Screens has been made.

A suggested procedure for submittal of methods and specifications to the editorial subcommittee has been accepted by the committee. The originating subcommittee chairman and at least one other member of that subcommittee will first review the proposed method with regard to the substance of the material and conformance with respect to the ASTM form. The drafts will then be reviewed for editorial form and nomenclature by Subcommittee VIII prior to submitting the method in its final form as a recommendation to the Society.

This report has been submitted to letter ballot of the committee, which consists of 65 members; 47 members returned their ballot, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. A. STANDING,
*Chairman.*

STANTON UMBREIT,
*Secretary.*

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### EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

*Revision of Tentative Specifications for:*

Nickel Alloy Cathode Sleeves for Electronic Devices (F 239 – 57 T)
Tungsten Wire Under 20 Mils in Diameter (F 288 – 54 T)
Round Wire for Use as Electron Tube Grid Laterals and Verticals (F 290 – 54 T)

The revision of Specification F 239 was accepted by the Standards Committee on December 13, 1957 and appears in the Compilation of ASTM Standards on Electron Tube Materials; the revisions of Specifications F 288 and F 290 were accepted on August 15, 1957, and appear in the 1957 Supplement to Book of ASTM Standards, Part 6.

# TECHNICAL PAPERS

## With Discussions

# CYCLIC STRAIN FATIGUE STUDIES ON AISI TYPE 347 STAINLESS STEEL*

By E. E. Baldwin,[1] G. J. Sokol,[1] and L. F. Coffin, Jr.[2]

## Synopsis

Constant temperature cyclic-strain fatigue tests are reported for AISI type 347 stainless steel at strain ranges of from 0.0035 to 0.020 in. per in. Parameters investigated included temperature (600 C max.), grain size, anisotrophy, and influence of sequential loading. Results are interpreted in terms of the plastic strain range – cycles to failure relationship $N^{\frac{1}{2}}\Delta\epsilon_p = c$.

The resistance of materials of construction to thermally induced stresses and strains is an important design factor in many applications. Examples include nuclear power plants and their rapid temperature changes during operation in response to power demands, and power producing equipment such as gas and high-temperature steam turbines where increased thermal transients are encountered. In heat transfer equipment, especially those subjected to rapid cyclic temperature transients, the design may be such that the thermal stresses and strains actually may exceed the yield point of the material. If this temperature change and resultant stress and strain are cyclic, the resistance of materials to low cycle fatigue becomes important.

Since AISI type 347 stainless steel has been used extensively in nuclear power plants as a structural material in parts subjected to cyclic temperature transients, there is considerable interest in its resistance to this type of loading. Experimental work was performed by Coffin (**1**),[3] in which the thermal stress fatigue resistance of AISI type 347 stainless steel was investigated using an apparatus which induced cyclic strain in a specimen by cycling its temperature while the specimen was constrained axially. By this method, a fatigue curve was established and various parameters determined for the material in which the controlling variable was cyclic strain induced by cycling the temperature of the specimen about some specified mean temperature.

Although this type of test gives a laboratory measurement of the resistance of a material to failure by repeated cyclic strains induced by cyclic temperature changes, it is not possible to separate the related effects of temperature and strain. It is desirable in many cases to have information on the cyclic strain resistance of materials at constant elevated temperature for practical reasons, as well as for a better understanding of the effect of both cyclic temperature and strain on the fatigue resistance of materials. Information of this kind is extremely sparse. Lui, Lynch, Ripling, and Sachs (2) and Lui (3) have performed low-cycle, high-strain fatigue tests on 24 ST aluminum. A comparison of thermal stress and constant temperature strain cycling together with an investigation of strain localizing effects was reported by Coffin (4). Coffin and Read (5) investigated the strain-softening of cold-worked AISI type 347 stainless steel using at a modification of the cyclic thermal stress apparatus referred to above. Some cyclic strain bend test results have also been reported (6, 7, 8).

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Knolls Atomic Power Lab., Atomic Energy Commission, General Electric Co., Schenectady, N. Y.

[2] General Electric Research Laboratory, Schenectady, N. Y.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 581.

It was evident that an extensive program, including the design and construction of new and special equipment, was required to explore the many important variables of the problem. These variables include the effect of cyclic strain, the resulting cyclic stress, cycles to failure, temperature, material composition, metallurgical structure (including grain size and anisotrophy) and sequential loading.

This paper outlines the apparatus, results obtained, analysis and interpretation of the results of tests on AISI type 347 stainless steel.

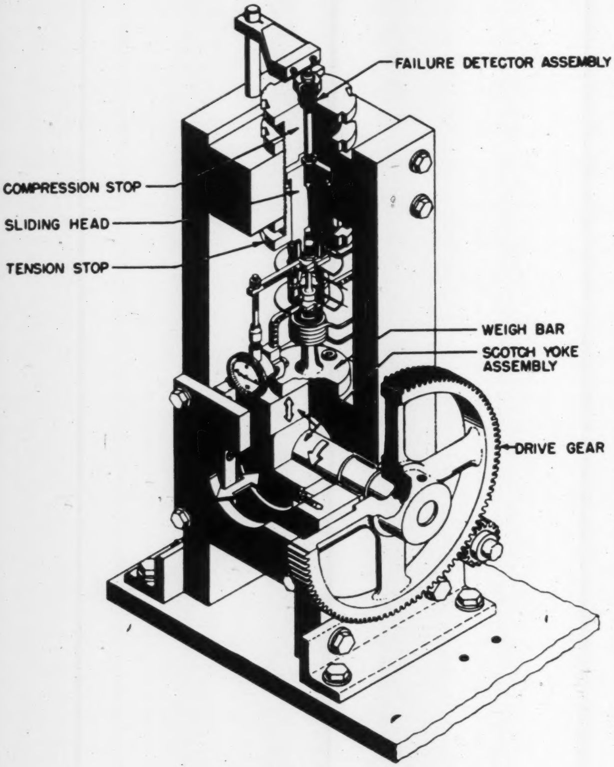

FIG. 1.—Strain Cycling Apparatus.

## TEST EQUIPMENT

In a conventional fatigue test, a specimen is subjected to a cyclic load to achieve a given stress, and the number of times this load can be applied to the specimen before it fails is determined. The strain fatigue test also determines the number of cycles to which a specimen can be subjected, but a controlled cyclic strain—not a stress—is applied to the specimen. The difference between the two methods of testing is of consequence when a substantial departure from linearity exists between stress and strain; this occurs when the number of cycles required for failure by fatigue is relatively low.

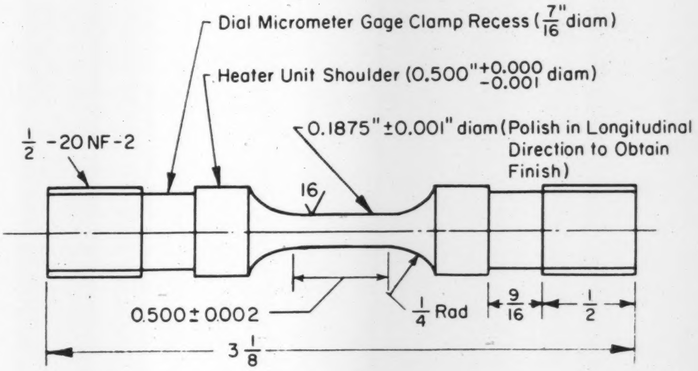
Dial Micrometer Gage Clamp Recess ($\frac{7"}{16}$ diam)
Heater Unit Shoulder (0.500" $^{+0.000}_{-0.001}$ diam)
$\frac{1}{2}$ -20 NF-2
0.1875" ±0.001" diam (Polish in Longitudinal Direction to Obtain Finish)
16
0.500 ± 0.002
$\frac{1}{4}$ Rad
$\frac{9}{16}$
$\frac{1}{2}$
$3\frac{1}{8}$

FIG. 2.—Strain Fatigue Specimen.

The cyclic-strain fatigue equipment developed for this investigation is shown in Fig. 1. Its essential function is to transmit a cyclic longitudinal strain to a test specimen (shown in detail in Fig. 2). This is done by oscillating the lower specimen fixture (weigh bar) through a constant vertical amplitude and by transmitting this motion through the specimen to the upper specimen fixture (called the sliding head). This fixture is actually a long piston, the amplitude of which can be adjusted by means of threaded stops as shown in Fig. 1. Thus, if the gap produced by the two stops exceeds the double amplitude of the lower fixture, the specimen merely oscillates with no strain; on the other hand when the gap is zero, the full amplitude of the lower fixture is transmitted to the specimen as cyclic strain.

To produce a linear cyclic motion of fixed amplitude in the lower fixture, a scotch yoke mechanism is employed. Circular motion is produced by an electric motor driving the scotch yoke assembly through a V-belt and stepped pulleys, reduction gear, and drive gear. The scotch yoke, consisting of an eccentric cam shaft, follower, and main block, transforms the rotary motion into a linear oscillatory motion of fixed amplitude.

The specimen train which is connected to the main block of the scotch yoke consists of a weigh bar, specimen, and sliding head. The weigh bar is an accurately machined steel bar upon which are mounted SR-4 type strain gages. When used with a strain indicator, these gages when calibrated can be used to determine the load and stress on the specimen during the cycle. The specimen, Fig. 2, is similar to a cylindrical tensile-type specimen with a test region of constant diameter accurately machined and polished gage section, and threaded ends. The test specimen is fastened to the sliding head, the motion of which, as pointed out above, can be arrested in any desired position of either the upward or downward stroke by means of the tension or compression stops and locking nuts.

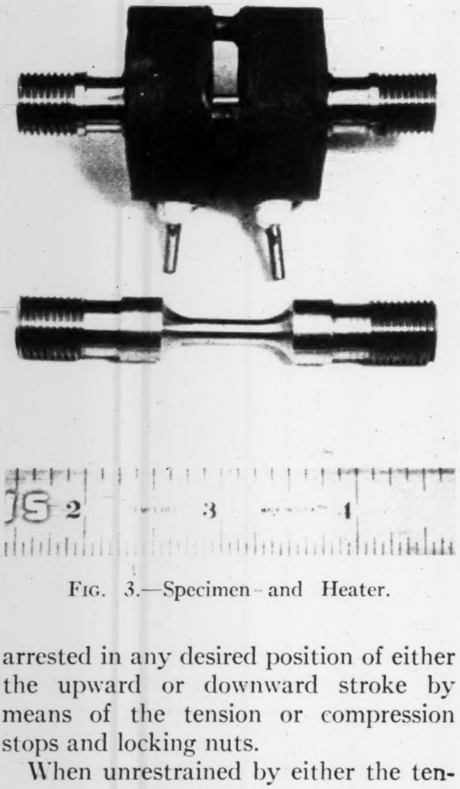

FIG. 3.—Specimen and Heater.

When unrestrained by either the tension or compression stops, the entire specimen train (scotch yoke, weigh bar, specimen, and sliding head) moves through an unconstrained linear oscillatory motion of about $\frac{1}{16}$ in. double amplitude or range. The motion of the sliding head is controlled by the position of the tension and compression stops. Since the gage length of the specimen is made the weakest part of the specimen train, the difference between the fixed and variable motion of the specimen train is imposed as mechanical strain in the gage length of the specimen. Knowing the range of the input motion, $X$, and the total strain range desired in the specimen, $\Delta\epsilon$, the tension and compression stops can be so adjusted to produce the desired range of head motion, $Y$, so that

$$\Delta\epsilon = \frac{(X - Y)}{L} \qquad \text{.............(1)}$$

where $L$ is the gage length. The relative positions of the tension and compression stops are set so as to produce a mean strain on the test specimen of zero. Thus, the strain amplitude is $\frac{\Delta\epsilon.}{2}$

The strain in the specimen during a test is actually measured by two dial gages fastened to a bracket, which in turn is fastened to the shoulders of the specimen shown schematically in Fig. 1. Hence, if there is any unequal strain in the specimen, the average strain may be determined by averaging the readings of both gages.

For elevated temperature tests, a 300 w resistance heater shown in Fig. 3 was used. It consists of two separate identical metal covered heater units connected together by thin tubular columns. The heater ends are located by a close tolerance fit over the shoulders at the ends of the specimen. The core of each heater is metal so that heat transfer is principally by conduction through the specimen shoulders and into the gage length of the specimen. This particular arrangement, where the gage length is heated by keeping the shoulders somewhat hotter than the gage length produces a very flat temperature gradient within the gage length and represents a very satisfactory solution to an otherwise difficult heating problem. The temperature of the specimen was determined by Chromel-Alumel thermocouples very lightly spot welded to the center of the gage length and connected to a thermocouple potentiometer controller.

To prevent overheating of the specimen fixtures and consequent damage to the dial gages and strain gages, heat shields and water cooling coils are fastened to the fixtures as shown in Fig. 1.

## MATERIAL

The material tested in this investigation was AISI type 347 stainless steel. Several material forms were used, depending upon the variable being investigated. In the determination of the effects of temperatures, grain size, and sequential loading, specimens were machined from $\frac{1}{2}$-in. diameter cold-drawn rod. To investigate the effect of anisotropy on strain fatigue strength, specimens were machined from seamless cold-drawn pipe, $5\frac{7}{8}$ in. inside diameter by $7\frac{1}{4}$ in. outside diameter. To investigate the effects of both grain size and anisotropy, specimens were machined from rolled and annealed plates $3\frac{1}{4}$ in. thick. The heat number and chemical composition of the above materials are listed in Table I.

TABLE I.—HEAT NUMBER AND COMPOSITION OF MATERIAL TESTED.

| Heat | Composition, per cent | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Carbon | Nickel | Chromium | Manganese | Silicon | Columbium | Tantalum | Phosphorus | Sulfur | Cobalt | Copper |
| 11615.. | 0.055 | 11.10 | 17.90 | 1.50 | 0.45 | 0.73 | 0.025 | 0.034 | 0.027 | 0.15 | 0.49 |
| X46941.. | 0.060 | 12.14 | 17.62 | 1.33 | 0.67 | 0.80 | .... | 0.016 | 0.009 | 0.07 | 0.06 |
| X14493.. | 0.05 | 10.48 | 17.40 | 1.63 | 0.49 | 0.71 | 0.08 | 0.030 | 0.011 | 0.14 | 0.24 |
| X14429.. | 0.05 | 10.70 | 17.96 | 1.44 | 0.54 | 0.74 | 0.03 | 0.028 | 0.012 | 0.07 | 0.22 |
| X15277.. | 0.06 | 10.72 | 17.24 | 1.62 | 0.52 | 0.63 | 0.08 | 0.022 | 0.010 | 0.10 | 0.33 |
| X15278.. | 0.05 | 10.86 | 17.36 | 1.62 | 0.24 | 0.46 | 0.08 | 0.035 | 0.008 | 0.10 | 0.31 |

Unless otherwise noted, all material was solution treated by heating to 1100 C and cooling rapidly.

## RECORDED AND CALCULATED DATA

During the conduct of strain fatigue tests certain data are obtained including:

$\Delta\epsilon$ = Total strain range per cycle,
$\Delta\sigma$ = Stress range per cycle,
$N$ = Number of cycles to failure, and
$T$ = Temperature of test.

With the above data and known properties of the material being tested, strain range can be separated into its elastic and plastic components (1):

$$\Delta\epsilon = \Delta\epsilon_{\epsilon} + \Delta\epsilon_{p}, \qquad (2)$$

where:

$\Delta\epsilon_{\epsilon}$ = Elastic strain range, and
$\Delta\epsilon_{p}$ = Plastic strain range.

The elastic strain range can be calculated by the relationship

$$\Delta\epsilon_{\epsilon} = \frac{\Delta\sigma}{E} \qquad (3)$$

where $E$ is the modulus of elasticity.

The values used for modulus of elasticity at the testing temperatures are based upon available published data.

The above data are presented graphically in three different ways. The total strain range can be plotted *versus* the logarithm of the cycles to failure, and the stress range can be plotted in the same manner. Since in strain fatigue the plastic strain range to which the specimen is subjected is the controlling variable (1), the logarithm of the plastic strain change can be plotted *versus* the logarithm of the cycles to failure.

In addition to the above data, the tensile stress-strain data are important

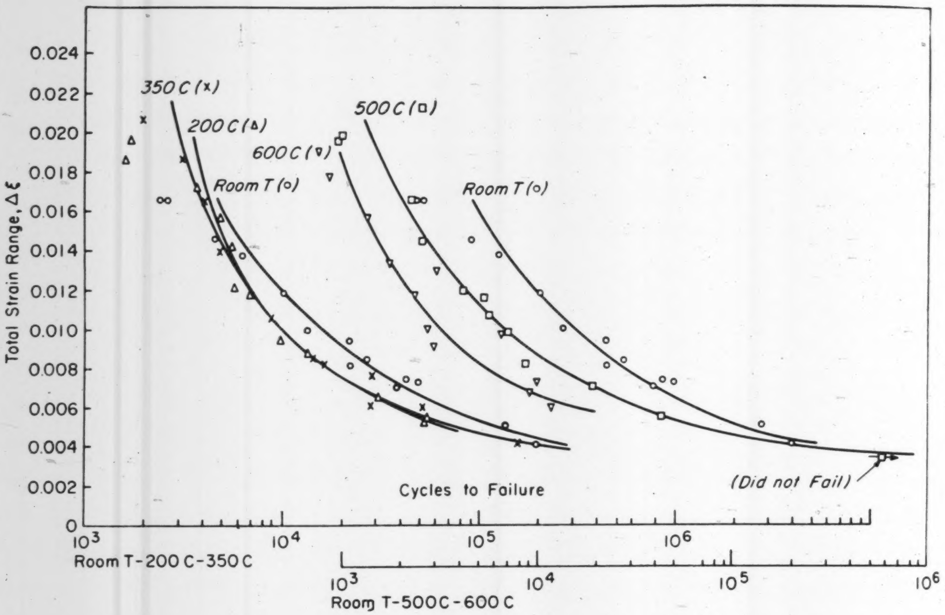

FIG. 4.—Total Strain Range *versus* Cycles to Failure 347 Stainless Steel Rod (Ht-11615).

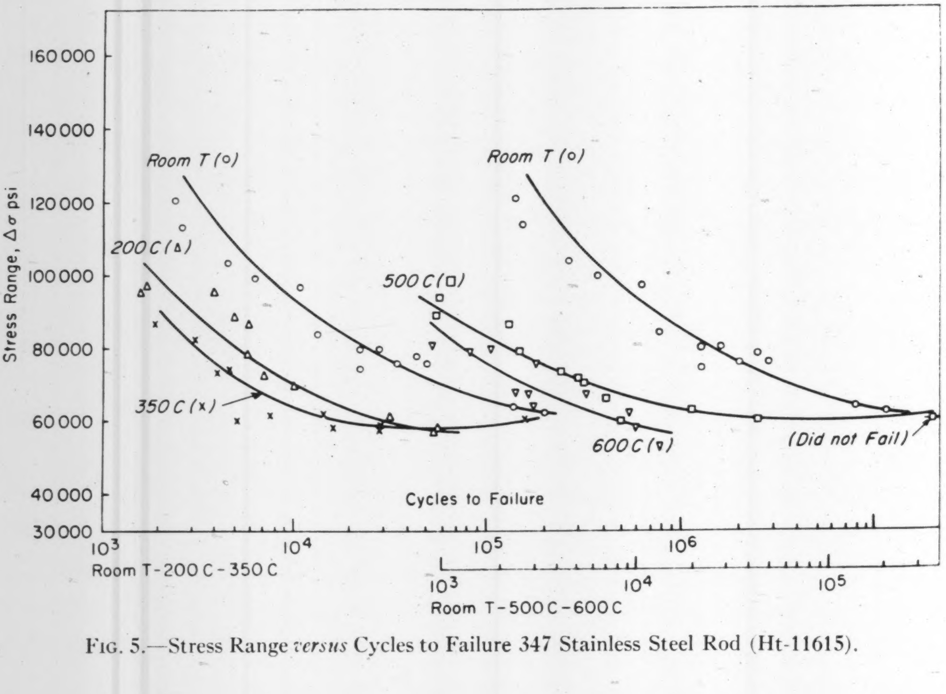

FIG. 5.—Stress Range *versus* Cycles to Failure 347 Stainless Steel Rod (Ht-11615).

for comparison and interpretation of the cyclic data. This information has been obtained for the test material and the same testing temperatures employed in the strain cycling tests.

## TEST RESULTS

### *Effect of Temperature:*

To determine the relationship between the cyclic strain fatigue resistance of type 347 stainless steel and temperature, strain fatigue and tensile on tests were conducted at room temperature, 200, 350, 500 and 600 C. The results of these tests are shown in Figs. 4 through 6.

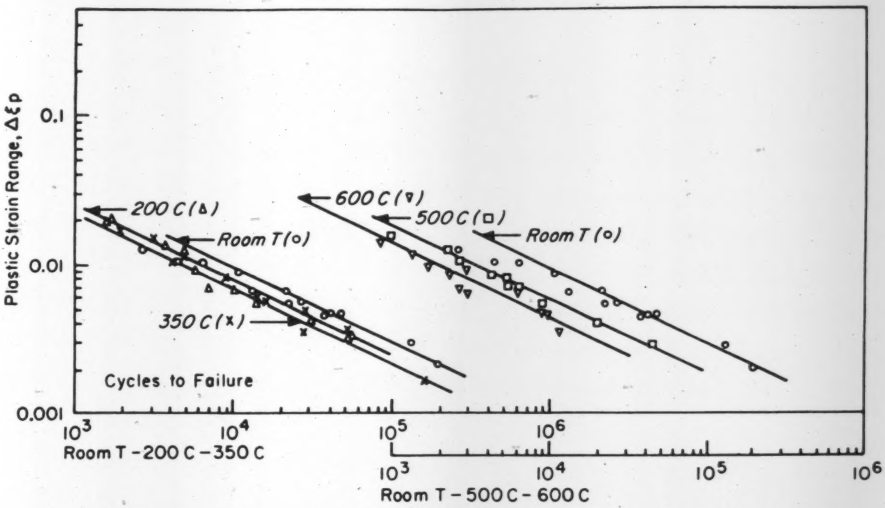

FIG. 6.—Plastic Strain Range *versus* Cycles to Failure 347 Stainless Steel Rod (Ht-11615).

Examination of these data shows that the total strain range *versus* cycles to failure, and stress range *versus* cycles to failure, and monotonic stress *versus* strain give families of curves which decrease with increasing temperature. An exception to this occurs at 500 C for the stress range (Fig. 5) and total strain range *versus* cycles to failure. In addition, examination of Fig. 6 reveals that the plastic strain range *versus* cycles to failure curves for both 200 and 350 C coincide.

It should be noted that for the highest stress levels, the various curves in Figs. 4, 5, and 6 are drawn somewhat above the test points. This is because of the buckling tendency of these particular tests. Buckling was particularly evident for the room temperature tests below $10^4$ cycles to failure. In Fig. 6 and, in fact, in all the subsequent figures involving plastic strain range and cycles to failure, straight lines having slopes of $-\frac{1}{2}$ are drawn through the various test points. The reasons for this will be considered at a later point.

### *Anisotropy:*

Since fabrication procedures may produce anisotropic effects in the material, and hence might influence the fatigue resistance of the material, tests were made on specimens machined from heavy-walled pipe and rolled plate. Possible anisotropic effects were determined by machining specimens parallel and perpendicular to the rolling or drawing direction of the material sample. The results of these investigations are plotted in Figs. 7 through 9.

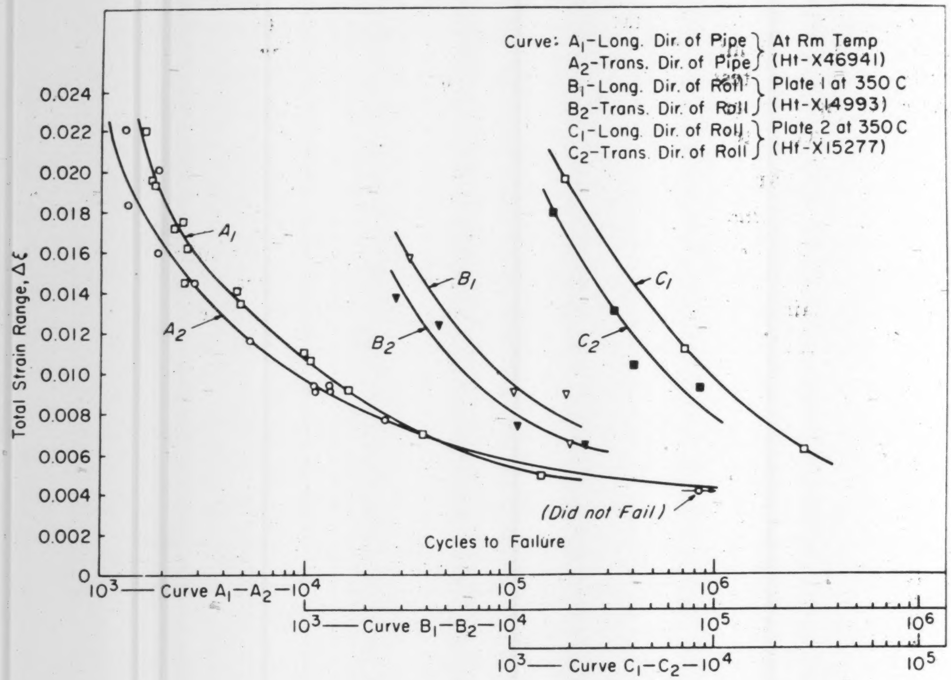

FIG. 7. Total Strain Range *versus* Cycles to Failure 347 Stainless Steel Seamless Pipe and Rolled Plate.

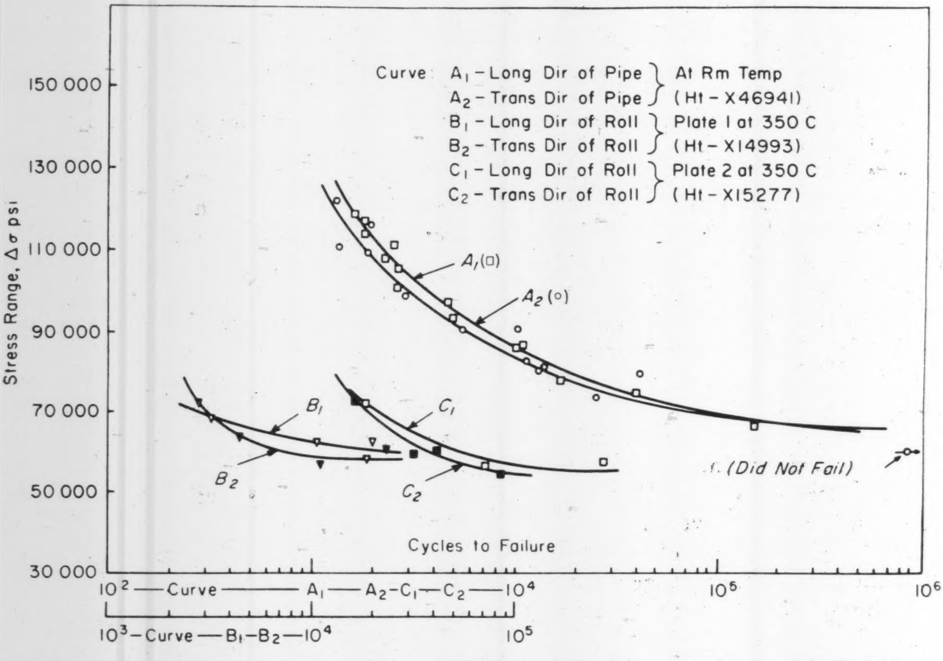

FIG. 8.—Stress Range *versus* Cycles to Failure 347 Stainless Steel Seamless Pipe and Rolled Plate.

Examination of these data indicates that there is some slight effect of anisotropy on the strain fatigue resistance. This is seen, for example, by comparing curve A₁ and A₂, B₁ and B₂, and C₁ and C₂ in Figs. 7, 8, and 9. In every case, the curves for specimens machined perpendicular to the fabrication direction are slightly lower than those for specimens machined longitudinal to the fabrication direction. There is a tendency, however, for the stress range curves to cross for the two plates tested.

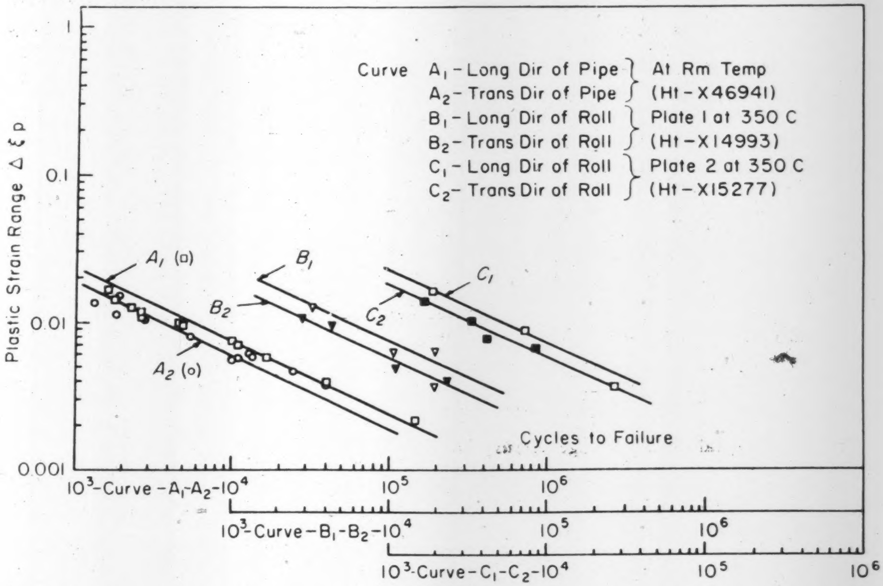

FIG. 9.—Plastic Strain Range *versus* Cycles to Failure 347 Stainless Steel Seamless Pipe and Rolled Plate.

TABLE II.—GRAIN SIZE STRAIN FATIGUE SPECIMENS.

| Material | Heat | Heat Treatment | ASTM Grain Size |
|---|---|---|---|
| Bar | 11615 | 30 min at 1050 C | 7 |
| Bar | 11615 | 5 min at 1100 C | 7 |
| Bar | 11615 | 30 min at 1300 C | 2 |
| Plate | X15278 | Annealed at 1070 to 1120 C | 1 to 5 |
| Plate | X15278 | Annealed at 1070 to 1120 C | 4 to 6 |

*Grain Size:*

The grain size of a metal affects such mechanical properties as tensile and creep strength. To investigate its effects on resistance to cyclic strain, two series of tests were run. Specimens machined from bar stock were given three different annealing treatments resulting in different grain sizes, and specimens were also cut from a $3\frac{1}{4}$-in. thick plate containing regions of two different grain sizes. Strain fatigue tests of both sets of specimens were run at 350 C. In Table II are summarized the micro structural characteristics of the two sets of specimens.

The results of this investigation are plotted in Figs. 10 through 12. Examination of these results seems to indicate that grain size has little or no effect upon

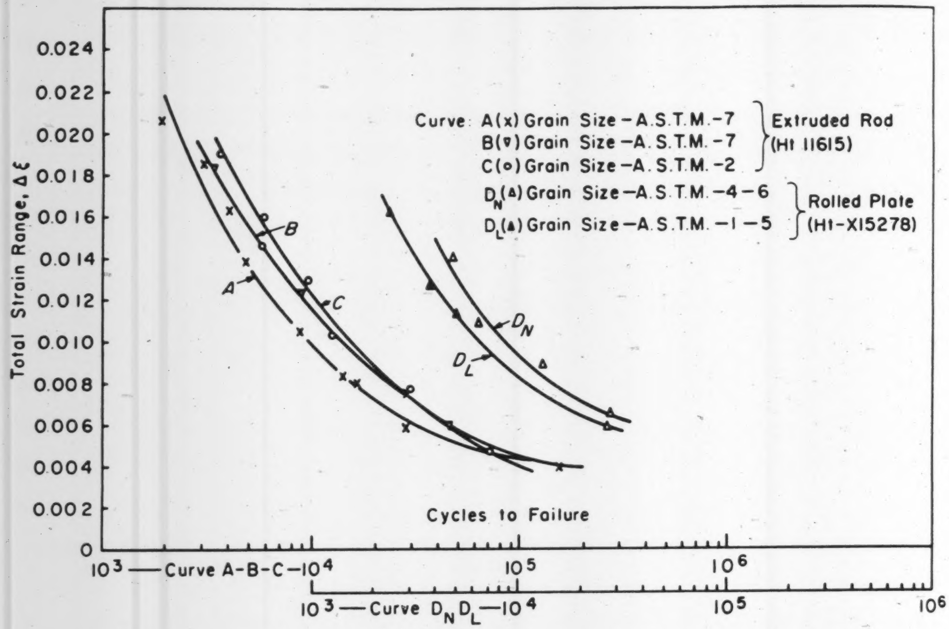

FIG. 10.—Total Strain Range *versus* Cycles to Failure of 347 Stainless Steel at 350 C.

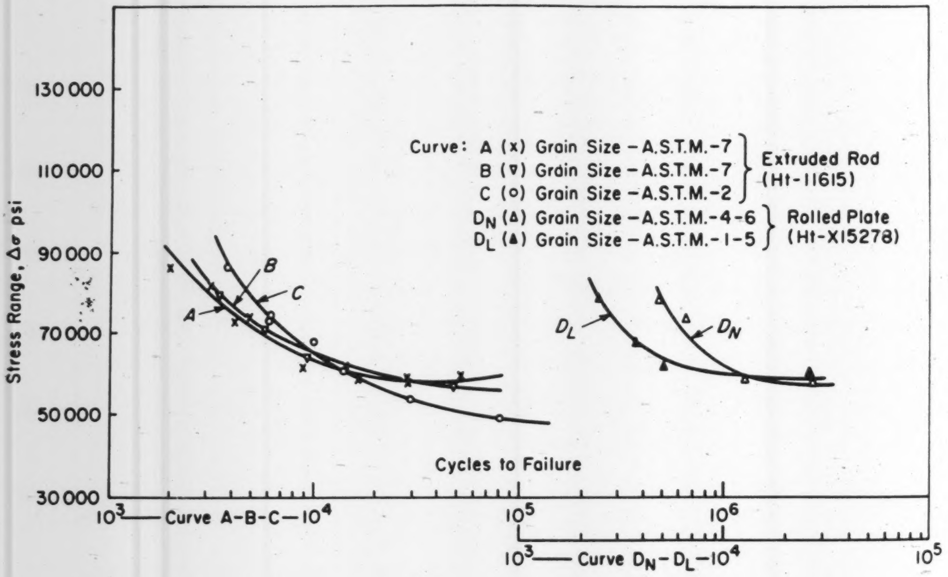

FIG. 11.—Stress Range *versus* Cycles to Failure 347 Stainless Steel at 350 C.

the strain fatigue resistance of 347 stainless steel. This is evidenced by the fact that for the plate material, the large grain size material had a higher fatigue strength than the small grained material, while for the bar stock specimens, the reverse was true.

*Sequential Loading:*

Since in service equipment, material will be subjected to cyclic applied strains of varying magnitudes, it was decided to determine what effect the application of strains of different magnitudes in specified sequence would have on the strain fatigue life of the material under investigation. To accomplish the above, four different test procedures were used.

*Test A.*—1000, 3000, or 6000 cycles at 1.0 per cent total strain range followed by cycling to failure at 0.6 per cent strain range.

*Test B.*—3000, 10,000, or 20,000 cycles at 0.6 per cent total strain range, followed by cycling to failure at 1.0 per cent total strain range.

*Test C.*—Repeat the following cycling sequence until failure; 1 cycle at 1.2 per cent total strain range, 2 cycles at 0.8 per cent total strain range and 17 cycles at 0.6 per cent total strain range.

*Test D.*—Alternate 1000 cycles at 0.6 per cent total strain range with 1000 cycles at 1.0 per cent total strain range until failure.

The percentage of the sequential life to that of a fixed strain range was calculated according to the relation

per cent of constant strain cycling life

$$= \frac{\sum_{i=1}^{n} N_i \Delta\epsilon\bar{p}_i^{2}}{c^2} \quad ..(4)$$

as given in reference (**9**), author's closure. Here $N_i$ and $\Delta\epsilon_{pi}$ are the number of cycles at a particular plastic strain range, and $c$ is a constant as discussed later. Equation 4 can be reduced to Miner's Eq 10 or

$$\text{per cent life} = \sum_{i=1}^{n} \frac{N_i}{N_{fi}} \times 100 \quad .....(5)$$

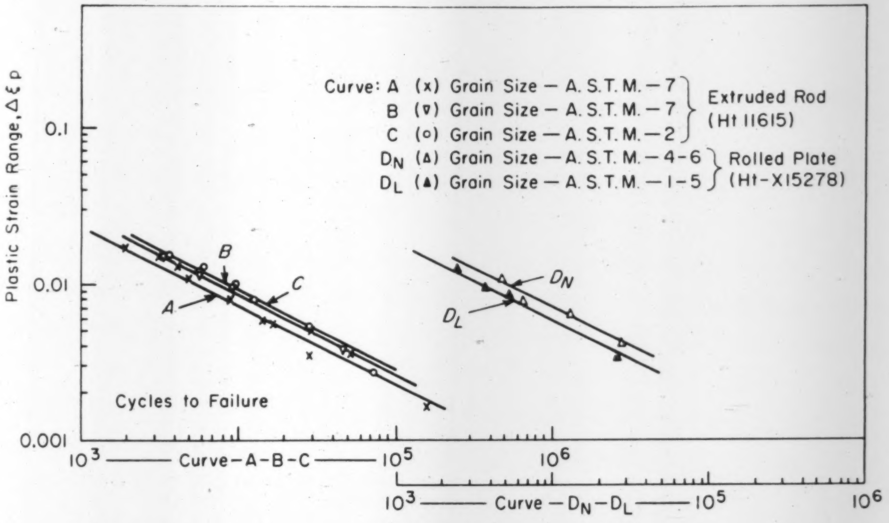

Fig. 12.—Plastic Strain Range *versus* Cycles to Failure 347 Stainless Steel at 350 C.

where $N_i$ is defined as above and $N_{fi}$ is the number of cycles for failure at the same strain range. Miner (**10**) proposed that for design purposes the summation in Eq 5 should equal 100 per cent. Data for $N_{fi}$ in Eq 5 were taken from Fig. 6.

TABLE III.—RESULTS OF SEQUENTIAL STRAIN CYCLING TESTS AT 350 C.

| Test | Strain Range, per cent | Stress Range, psi | Cycles $(N)i$ | Percentage of Strain Cycling Life[a] |
|---|---|---|---|---|
| A....... | 1.05 | 63 300 | 1 000 | 99 |
| | 0.60 | 59 200 | 30 544 | |
| A....... | 1.02 | 63 300 | 3 000 | 97 |
| | 0.64 | 59 200 | 17 117 | |
| A....... | 1.00 | 66 500 | 6 000 | 122 |
| | 0.64 | 59 200 | 15 486 | |
| B....... | 0.61 | 59 600 | 3 000 | 154 |
| | 1.03 | 67 200 | 11 300 | |
| B....... | 0.60 | 56 400 | 10 000 | 144 |
| | 1.04 | 66 800 | 9 056 | |
| B....... | 0.60 | 60 000 | 20 000 | 112 |
| | 1.06 | 69 200 | 4 322 | |
| C....... | 1.19 | 78 200 | 1 509 | 88 |
| | 0.77 | 69 500 | 2 536 | |
| | 0.60 | 67 400 | 21 350 | |
| C....... | 1.21 | 70 000 | 1 449 | 112 |
| | 0.81 | 65 600 | 2 888 | |
| | 0.60 | 60 700 | 23 934 | |
| D...... | 0.61 | 53 600 | 13 000 | 199 |
| | 1.00 | 61 500 | 12 900 | |

[a] Calculated on basis of tests at constant strain range.

## DISCUSSION

The above described results provide information on the resistance of AISI 347 stainless steel to constant temperature strain cycling under a variety of testing and structural conditions. As mentioned earlier, there are a considerable number of examples of cyclic-strain induced loadings occurring in engineering structures, and the design under these conditions must be based on fatigue data obtained by strain cycling rather than stress cycling. This is particularly true for finite cycle design where a limited number of severe cycles are expected and are incorporated in the design. Under these circumstances a considerable degree of non-linearity exists between cyclic stress and cyclic strain, and difficulties and uncertainties will result if cyclic stress test results are applied to cyclic strain problems. For example, it is not uncommon to encounter design stresses of thermal origin of the order of 100,000 psi (when calculated elastically) where the tensile strength of the material is 60,000 psi. Under these circumstances a design based on stresses has little meaning, whereas the equivalent total strain range or the plastic strain range is a more natural basis for assessing the cyclic life of the structure. Hence the need for cyclic strain fatigue information becomes evident.

In the above results, the stress range, total strain range, and plastic strain range have been represented against the cycles required for failure. The stress range is, of course, the measured stress but bears no direct relationship to an elastically calculated stress because of the high degree of non-linearity between cyclic stress and strain. The total strain range is a more directly usable quantity, since it is often possible to relate this quantity to elastically calculated strains and thus predict fatigue failure (**9**). On the other hand, the plastic strain range, $\Delta\epsilon_p$, is felt to be a fundamentally significant variable in predicting fatigue failure. In reference (**1**) and (**6**) the importance of this quantity was considered. It was shown there that the relationship

$$N^k \Delta\epsilon_p = c \ldots\ldots\ldots\ldots\ldots (6)$$

where $k \approx \frac{1}{2}$ and $c$ was a constant related to the fracture ductility, accurately predicted the known strain cycling data available. It is therefore of considerable interest to see if this equation represents the present results. Two comparisons can be made. The first relates to the constancy of the slope ($-\frac{1}{2}$), the second compares the actual ductility for a particular temperature and structure with that predicted by Eq 6. For the former comparison it is merely necessary to draw a straight line of slope equal to $-\frac{1}{2}$ through the test data on a plot of log $\Delta\epsilon_p$ *versus* log $N$. This comparison is seen in Figs. 6, 9, and 12.

To observe the degree to which Eq 6 predicts the fracture ductility, it should be pointed out first that if failure occurs in one-quarter of a cycle, the plastic strain range so produced is the fracture ductility (represented as the true strain at fracture, $\epsilon_f$). When this is introduced into Eq 6, the quantity $c$ becomes

$$c = (1/4)^{\frac{1}{2}}\epsilon_f = \frac{\epsilon_f}{2} \quad \text{or} \quad 2c = \epsilon_f \quad \ldots.(7)$$

To make the comparison, it is necessary merely to determine the value of $2c$ in Eq 6 for a particular $N$ and $\Delta\epsilon_p$ (using $k = \frac{1}{2}$) and compare this with the quantity $\epsilon_f$ obtained in a separately conducted tension test under the same conditions of temperature and structure. This comparison is made in Table IV.

TABLE IV.—COMPARISON OF CONSTANT $2c$ IN EQ 6 WITH FRACTURE DUCTILITY.

| Shape | Direction | ASTM Grain Size | Temperature, deg Cent | $2c$ | $\epsilon_f$ [a] | $\frac{2c - \epsilon_f}{2c} \times 100$, per cent |
|---|---|---|---|---|---|---|
| Rod | Long | 7 | R.T. | 1.90 | 1.32 | 31 |
| Rod | Long | 7 | 200 | 1.56 | 1.30 | 17 |
| Rod | Long | 7 | 350 | 1.40 | 1.03 | 27 |
| Rod | Long | 7 | 500 | 1.18 | 1.09 | 8 |
| Rod | Long | 7 | 600 | 0.86 | 1.13 | −31 |
| Plate Heat 14993 | Long | 4 to 6 | 350 | 1.46 | 1.20 | 18 |
| Plate Heat 14993 | Trans | 4 to 6 | 350 | 1.16 | 0.59 | 49 |
| Plate Heat 15277 | Long | 4 to 6 | 350 | 1.46 | 1.06 | 27 |
| Plate Heat 15277 | Trans | 4 to 6 | 350 | 1.16 | 0.59 | 49 |
| Pipe | Long | 7 | R.T. | 1.50 | 1.20 | 20 |
| Pipe | Trans | 7 | R.T. | 1.20 | 1.11 | 8 |
| Plate | Long | 4 to 6 | R.T. | 1.40 | 1.19 | 15 |
| Plate | Long | 1 to 5 | R.T. | 1.18 | 1.26 | −7 |
| Rod | Long | 7 | 350 | 1.84 | 1.11 | 40 |
| Rod | Long | 2 | 350 | 1.70 | 1.18 | 31 |

[a] $\epsilon_f = \ln \frac{A_0}{A}$, where $A_0$ is the initial cross-sectional area and $A$ is the final cross-sectional area.

One may conclude from the examination of the fit of the test data to $k = \frac{1}{2}$ and the agreement between the predicted and experimental ductility data Eq 6 very closely predicts the test results obtained. Since only two decades of cycles to failure are covered in the present tests, it is difficult to determine accurately the best slope of the data, and the slope of $-\frac{1}{2}$ fits as well as any that might be drawn. This might be questioned, for example in Fig. 9; however a greater spread in strain range is required before this point can be answered completely.

With respect to the comparisons of ductility, Table IV, it will be observed that temperature has a more pronounced effect on the quantity $2c$ than on the fracture ductility. For example, at room temperature $\epsilon_f$ is some 30 per cent less than $2c$, while at 600 C it is 31 per cent greater than $2c$. While the reason for this is not known with any certainty, it is suspected that time-dependent effects must be reckoned with. For example the total time at temperature for the tension tests and the strain cycling tests were substantially different, this difference increasing with decreasing plastic strain ranges. This could introduce a variety of effects such as oxidation, grain boundary precipitation, grain boundary voids, etc., not accounted for in the short-time tests.

Another difference found from Table IV is the apparent insensitivity of the effect of orientation and grain size on the value of $2c$ in comparison with $\epsilon_f$. Without further knowledge as to the relationship between fracture ductility and strain cycling fatigue failure, it is difficult to account for this or the other various differences observed. It may be, for example, that the relationship is purely fortuitous. However, the engineering implications of such a relationship are quite important and further study should be given to the problem. In so far as the present tests are concerned, it may be said that an analysis based on using the fracture ductility appears to be conservative by about 30 per cent on the average, except where temperatures above 500 C are encountered.

With respect to the stress cycling results, it would be expected that the behavior of the stress range with cyclic strain can be determined qualitatively from the monotonic stress-strain curves. An exception to this appears to be the 500 C results indicated in Figs. 4, 5, and 6. In Figs. 4 and 5 particularly, the 500 results do not fit in with the family of curves produced by the other temperatures. This is attributed to some metallurgical factor, as yet undetermined. The effect has been found before in the strain cycling of cold-worked metals (5) and in thermal cycling (11).

The insensitivity of variations of grain size and anisotropy on the cyclic strain fatigue is not too surprising, in view of the effects of these parameters generally encountered in stress cycling.

## SUMMARY

Test results are reported for AISI type 347 stainless steel subjected to constant-temperature cyclic strains ranging from 0.0035 in. per in. to 0.02 in. per in., where pronounced non-linearity between cyclic stress and cyclic strain occurs, and where failures result in from $10^3$ to $5 \times 10^5$ cycles. Parameters of the testing program include temperature, grain size, anisotropy, and sequence of loading. These results are pertinent for those applications where structures are subjected to strain-induced cyclic deformation.

Results are given in terms of the stress range, total strain range, and plastic strain range. Good agreement is found when applying the previously determined relationship $N^{\frac{1}{2}} \Delta\epsilon_p = c$, both for the exponent $\frac{1}{2}$ (which is the negative of the slope on a log-log representation of these variables) and the constant $c$ (which is compared to one-half the fracture ductility in simple tension).

When considering the above relationship, neither grain size nor anisotropy appear to have a significant effect. On the other hand, the temperature parameter has a pronounced effect on the constant $c$ which is not revealed from the fracture ductility.

Damage produced by sequential loading was determined by a modification of Miner's relationship for comparison with tests conducted by single cyclic strains. Life in sequential loads varied from 72 to 163 per cent of the life found in the simple tests.

## REFERENCES

(1) L. F. Coffin, Jr., "A Study of the Effects of Cyclic Thermal Stresses on Ductile Metals," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 76, pp. 931–950 (1954).

(2) S. I. Lui, J. S. Lynch, E. J. Ripling, and G. Sachs, "Low Cycle Fatigue of Aluminum Alloy 24 ST in Direct Stress," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 175, p. 469 (1948).

(3) S. I. Lui, "Effects of Precompression on the Behavior of 24 ST 4 During Cyclic Direct Stressing," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 178, pp. 452 (1951).

(4) L. F. Coffin, Jr., "The Problem of Thermal Stress Fatigue in Austenitic Steels at Elevated Temperatures," Symposium on Effects of Cyclic Heating and Stressing on Metals at Elevated Temperatures, Am. Soc. Testing Mats. (1954). (Issued as separate publication *ASTM STP No. 165*.)

(5) L. F. Coffin, Jr. and J. H. Read, "A Study of the Strain Cycling and Fatigue Behavior of a Cold Worked Metal," International Conference on Fatigue of Metals, London, Sept., 1956.

(6) A. C. Low, "Short Endurance Fatigue," International Conference on Fatigue of Metals, London, Sept. 1956.

(7) A. Johansson, "Fatigue of Steels at Constant Strain Amplitude and Elevated Temperature Proceedings," Colloquium on Fatigue, Stockholm, Julius Springer, Berlin, 1956.

(8) J. H. Gross and R. D. Stout, "Plastic Fatigue Properties of High-Strength Pressure-Vessel Steels," *Welding Journal* (*Research Supplement*), Vol. 34, p. 161–5 (1955).

(9) L. F. Coffin, Jr., "Design Aspects of High Temperature Fatigue with Particular Reference to Thermal Stresses," *Transactions*, Am. Soc. Mechanical Engrs. Vol. 78, pp. 527–532 (1956).

(10) M. A. Miner, "Cumulative Damage in Fatigue," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 67, pp. 159–164 (1945).

(11) L. F. Coffin, Jr., "An Investigation of Thermal Stress Fatigue as Related to High Temperature Piping Flexibility," *Paper No. 56-A-178*, Am. Soc. Mechanical Engrs. (1956).

## DISCUSSION

MR. L. F. KOOISTRA[1] (*presented in written form*).—The data presented in the paper by Messrs. Baldwin, Sokol, and Coffin indicate a very simple and valuable relationship between plastic strain and cyclic life. By separating elastic and plastic strain, amazingly consistent data have been obtained. Since some data in the plastic range are also available on pressure vessel steels,[2] it is of general interest to see how the behavior of these materials compares with the data presented in the paper on stainless 347. The plastic strains on the most ductile (ASTM Specification[3] A 201, grade A steel) and the strongest (T-1) steel were plotted in comparison with the curve for room temperature of Fig. 6 of the paper. The plastic strain was obtained by subtracting the yield point strain from the total strain. There undoubtedly is some discrepancy in this procedure because the elastic limit of these materials can be expected to decrease somewhat after several cycles of reversed plastic strain. Correction for such strain softening may shift upward a small amount the curves shown in the accompanying Fig. 13. Although the two testing methods are quite different, there appears to be very good qualitative agreement in the data on the two types of materials tested.

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[1] The Babcock & Wilcox Co. Research Center, Alliance, Ohio.

[2] L. F. Kooistra, "Effect of Plastic on Pressure-Vessel Materials and Design," *The Welding Journal*, March, 1957.

[3] Tentative Specification for Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 54 T), 1956 Book of ASTM Standards, Part 1, p. 487; Specification for Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 – 56), 1956 Book of ASTM Standards, Part 1, p. 566.

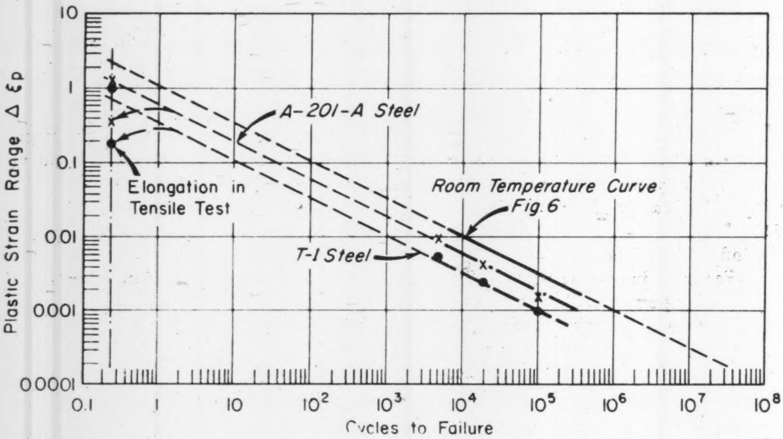

FIG. 13.—Plastic Strain *versus* Cycles to Failure Comparison of PVRC Data on Pressure Vessel Steels with 347 Stainless Steel Data.

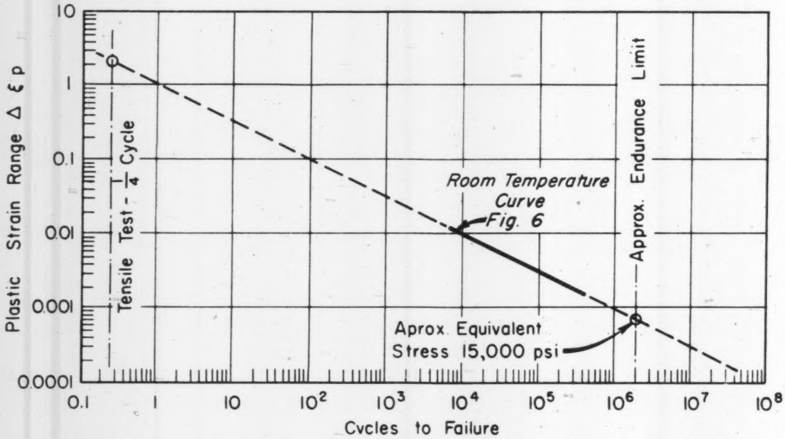

FIG. 14.—Plastic Strain *versus* Cycles to Failure 347 Stainless Steel Rod Curve of Fig. 6 Extrapolated.

Although so far only 2 decades have been covered, it is interesting to speculate on the extreme limits of the equation $N^k\Delta\epsilon_p = C$ by extending the room temperature curve of Fig. 6 from the $\frac{1}{4}$ cycle of the tension test to 2,000,000 cycles and over for the conventional endurance limit as shown in Fig. 14. On the left side of the plot, this extension would lead to a strain range $\Delta\epsilon_p$ of about 2.2. This is obviously too high by about a factor of 2. Although this discrepancy is not too serious for an extrapolation over such a wide range, it does indicate that when it comes to choosing a simple test for the purpose of characterizing a material with respect to plastic fatigue, it would be more accurate to conduct a singular low cycle fatigue test in preference to a conventional tension test.

There is another good reason for this choice in the fact that it takes several plastic cycles before the shape of the load-strain hysteresis is stabilized. The limited experience in testing specimens at large plastic strains also indicates that the elastic behavior with respect to strain softening or Bauschinger effect can only be established after the material has been subjected to several strain reversals. It is even suspected that the modulus of elasticity undergoes a change (decrease) during the initial cycles which would change the magnitude of the elastic part of the total strain.

With the proper testing equipment available, such a low cycle fatigue test of a few thousand cycles would not be much more expensive or time consuming than a conventional tension test, particularly, if both have to be conducted at elevated temperatures.

The right hand extrapolation of the curve shows that the endurance limit strain at 2,000,000 cycles and over is by no means purely elastic. Indications are that considerable plastic action takes place even when the cyclic life enters the region of infinite life.

The cyclic strain relationship discovered by the authors is extremely pertinent and useful in predicting the fatigue life of structures. It will be interesting to see how well it applies to other materials.

Since an important application of this relationship is in the field of severe thermal strains, further work will be necessary to determine the behavior of materials when subjected to relatively large equibiaxial strains.

MR. HERBERT T. CORTEN.[4]—I will confine my comments to the results shown in Table III for the sequential loading tests.

It is interesting to note that these data exhibit trends similar to those found in the literature for experiments carried out at room temperature. That is, when the stress (or strain) is completely reversed and the high stress is applied first (test A), the summation of cyclic ratios is found to be less than one. Conversely, when the low stress is applied first (test B), the summation of cyclic ratios is often found to be greater than one. The type C load sequence is of particular significance from the point of view of service performance. Again, the trend of the summation of cyclic ratios, $\sum \frac{n}{N} < 1$, agrees with the data in the literature for completely reversed stress room temperature experiments. The present data are limited to a total of nine specimens. Considering the scatter associated with fatigue, even low-cycle fatigue, it appears fortuitous that the data give such consistent trends.

Extrapolating from the trends found at room temperature, the value of the summation of cyclic ratios may increase if (*a*) a notch were introduced or (*b*) the range of stress were changed to include a high mean stress. Information concerning these two points would be of considerable interest; consequently, it is hoped that the authors will further pursue this phase of the investigation.

MR. D. D. ROSARD.[5]—Figures 4, 5, and 6 of the paper give the total strain range, the stress range and the plastic strain range *versus* cycles to failure. The first question is: if you maintain the total strain range constant throughout the test, have you noticed whether the stress range remains nearly constant, resulting in a constant plastic strain range throughout the length of the test?

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The second question is: total strain range is plotted on a semi-log type of plot, the plastic strain range on a log-log plot. Is there any particular significance to this difference?

MR. G. M. SINCLAIR.[6] (*by letter*).—The authors have presented a most important and interesting set of results and analyses. It seems strange that relatively little work has been done in the field of strain cycling fatigue when the preponderance of evidence has, for many years, shown that fatigue is essentially a problem of plastic deformation. There are two points relating to the work presented which I find of especial interest. First, in developing the expression $N^k \Delta\epsilon_p = C$, the authors mention that the slope in the log-log plot $k$ is equal to $-\frac{1}{2}$ and that if the straight-line portion is extrapolated back to $N = \frac{1}{4}$ (single stroke tension failure) the true strain at fracture falls on the line for the remainder of the cyclic fatigue data. Since in ductile metals, such as were used here, strains of this order are invariably accompanied by necking, it would appear that the state of stress at the time of failure would be quite different from that under fatigue conditions. In other words, in the tension test, the failure would be expected to initiate in a region of hydrostatic tension while this is not the case under cyclic straining and any correlation might then be fortuitous. Secondly, some years ago, a study was made of the influence of grain size on the work-hardening and fatigue characteristics of 70-30 brass.[7] As a portion of this work, numerous true stress - natural strain measurements were made and analyzed. It was found that fracture of the specimen occurred at approximately the same true stress irrespective of the state of the material, whether initially cold-worked or dead soft. The total work done in plastically deforming the material to fracture was found by graphical integration of the stress-strain diagrams and it appeared that the work required to fracture the material was also independent of the initial state. In view of this, I should like to ask the authors if they feel that failure due to repeated cyclic straining represents an attainment of some limiting amount of plastic work which may, in some way, account for the slope of $-\frac{1}{2}$ in their strain-fatigue relationship which also appears to be independent of the initial state of the material.

MESSRS. E. E. BALDWIN, G. J. SOKOL, L. F. COFFIN, JR. (*authors' closure*).—The authors would like to thank Messrs. Corten, Sinclair, Rosard, and Kooistra for their interesting and pertinent discussions to this paper. Mr. Corten has commented on the agreement in trend between the sequential loading tests reported in the present paper and those reported in the literature where completely reversed cyclic stress is the controlling variable. Although a limited number of tests have been conducted using sequential cyclic strain rather than cyclic stress, the authors are of the opinion that the consistent trends found are not entirely fortuitous. It should be noted that considerable care was taken in preparing the test specimens so as to minimize the influence of surface conditions. Of more importance, however, is the fact that these tests were conducted at levels of cyclic strain in which the entire gage length of the specimen was subjected to reversed plastic strain. This has the effect of reducing the degree of selectivity that normally accompanies fatigue failure after a large number of cycles where only small regions of the entire structure are subjected to plastic deformation. As a result, the scatter band for cycles to failure is considerably narrowed and more nearly corresponds to the type of scatter one would experience in obtaining the fracture ductility of a ductile material. No further work of this type is being contemplated at the present. However, the authors would like to point out that it is not feasible to conduct a low-cycle fatigue test using cyclic strain as the controlling variable and keeping the mean strain zero while at the same time imposing a high mean stress. Some reflection on this situation would reveal that under the strain conditions imposed the high mean stress would immediately relax. Should the mean stress be constant, the mean strain will no longer remain at zero but will change in the direction of the applied stress.

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[7] G. M. Sinclair and W. J. Craig, "Influence of Grain Size on Work Hardening and Fatigue Characteristics of Alpha Brass," *Transactions*, Am. Soc. Metals, Vol. 44, pp. 929–948 (1952).

Mr. Sinclair has raised two very interesting points with respect to the interpretation of the results given in the paper. It is indeed true that the state of stress under conditions of failure in a tension test is quite different from that occurring in a fatigue test. It is also true that the metallurgical structure at the time of failure is quite different between these two tests. Nevertheless, it can be shown that for a wide variety of materials of structural importance, the fracture ductility can be determined rather well by extrapolating the relationship $N^{\frac{1}{2}}\Delta\epsilon_p = C$ to $N = \frac{1}{4}$.[8] This fact is of considerable significance in engineering design where cyclic strains are of importance.

With respect to Mr. Sinclair's second and related point, it is certainly true that prior cold work, in general, does not bear a simple relationship to the fracture ductility as determined in a tension test. The relationship would obviously be more direct if the method of producing the prior cold work was identical to that used to obtain the ductility. Thus, in studying the effect of prior cold work one should perhaps introduce the cold work by prior tension if true strain at fracture is to be used as the measure of ductility. Studies of this sort have recently been carried out at the General Electric research laboratory and indicate that when prior cold work is introduced by tension the subsequent fracture ductility does fit the cyclic strain fatigue data quite well. On the other hand, agreement is nowhere near as good when the prior cold work is a result of some more conventional cold-working operation such as swaging.

Mr. Sinclair has suggested that failure from repeated cyclic straining may be the result of the attainment of a limiting amount of plastic work. If this is indeed so, it does represent another way of expressing the result obtained. However, it is the authors' feeling that although this concept is indeed an interesting one, it introduces no new insight into the interpretation of the behavior of materials under cyclic straining since the same questions can be asked as to the reasons for such a limit as can be asked regarding the expression $N^{\frac{1}{2}}\Delta\epsilon_p = C$.

In answer to Mr. Rosard's question concerning the behavior of the stress range during the test and its effect on the plastic strain range, it should be pointed out that the total strain range was maintained constant during the course of the test. Since materials may harden or soften as a result of cyclic plastic strain, the stress range will then change during the test. Unless the strain range chosen is extremely high, the hardening or softening effects are not appreciable such that the elastic strain range and, as a consequence, the plastic strain range remains relatively fixed. Although the results were obtained by using an average stress range for any one test, no significant difference would be found had the total strain range been continually corrected so as to maintain a constant plastic strain range.

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[8] See for example, L. F. Coffin, Jr., "The Resistance of Materials to Cyclic Thermal Stresses," Fourth Sagamore Ordnance Materials Research Conference, Aug., 1957.

As to Mr. Rosard's second question relating to the plotting of the total strain range *versus* cycles to failure on a semi-log basis and the representation of the plastic strain range on a log plot it should be pointed out that if both quantities were represented by a log-log representation, little difference would be noted when the levels of strain are sufficiently high ($\Delta\epsilon > 0.02$). However, when the strain level is such that the elastic strain range becomes large in relation to the plastic strain range, considerable deviation occurs between the plastic and total strains and the total strain range is no longer linear with respect of cycles to failure on the log-log representation.

The authors are indebted to Mr. Kooistra for supplying additional information for the ASTM Specification A 201, grade A steel and A 302 grade B steel[3] represented in terms of the plastic strain. He indicates that for these two materials, the fracture ductility values plotted at $\frac{1}{4}$ cycle on Fig. 6 do not fit the cyclic strain data particularly well. It can be shown that a large part of the discrepancy that arises here is due to the definition of ductility. In the authors' paper the true strain at fracture is used as the value for the plastic strain range at $\frac{1}{4}$ of a cycle while Mr. Kooistra is apparently using a ductility based on per cent elongation. The authors have taken the liberty of referring to Mr. Kooistra's paper[2] and have converted the ductility values given in his Table I to the true strain at fracture. For the steel A 201-A having a per cent elongation of 35.5 per cent and a per cent reduction of area of 68.2 per cent, the true strain at fracture becomes 0.99. For A 302 grade B steel having a per cent elongation of 17.5 per cent and a per cent reduction in area of 63.2 per cent, the true strain of fracture is 1.14. With reference now to Fig. 13, it will noted be that these two ductility values when plotted at $N = \frac{1}{4}$ cycle fit very closely to the extrapolated cyclic strain curves drawn by Mr. Kooistra for these two steels.

Despite the fact that the agreement in fracture ductility and the cyclic strain data now appears to be quite favorable the authors agree with Mr. Kooistra that cyclic strain tests should still be used when determining the resistance of materials subjected to cyclic strain fatigue.

# THE FATIGUE BEHAVIOR OF DECARBURIZED STEEL*, †

By H. A. Lipsitt[1] and G. T. Horne[2]

## Synopsis

Fatigue tests in completely reversed axial tension-compression have been made on a mild steel (in the hot-rolled condition) and on specimens of this steel after decarburization. Further, rotating-beam (R. R. Moore) fatigue tests have been made on the decarburized material in an effort better to define the fatigue limit behavior of this very low-carbon (approximately 0.003 per cent) material.

The data on the materials (as received and as decarburized) are discussed in the light of a strain aging explanation to account for their difference.

The phenomenon of the fatigue limit has puzzled scientists since the time of James and Galton (**1**),[3] Fairbairn (**2**), Wöhler (**3**), and Bauschinger (**4**). Moore and Kommers (**5**) in 1921 stated that the sharp break in the *S-N* curve meant that there was a distinct and sudden change in fatigue behavior at this point. Later, Moore and Jasper (**6**) postulated that in fatigue we are, in reality, observing the summation of two distinct and opposing types of behavior. They conceived of the *S-N* curve as the summation of the strengthening action of cold working under the applied stress and the destructive action of crack formation and propagation. While this explanation may be qualitatively correct in that there are two opposing effects, the two effects selected cannot be those responsible, for it is obvious that the processes they described fit the case of non-ferrous materials as well—most of which do not show a fatigue limit of the same type as mild steel.

The explanation of the fatigue limit must depend on one or more differences between "ferrous" and "non-ferrous" materials; there are two obvious differences between materials that do not exhibit this sharp fatigue limit behavior (non-ferrous) and those that do. Nearly all materials showing the true fatigue

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† This paper represents a portion of a thesis by Harry A. Lipsitt, submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy at the Carnegie Institute of Technology.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 598.

limit behavior exhibit either "peculiar" slip behavior in the parent metal (7) or strain aging effects in the alloy or both. An investigation of the slip behavior of single crystals of iron under fatigue loading has already been reported (8). Removing (or vastly reducing) the yield point and strain aging effects in iron was the approach used in the present investigation.

## LITERATURE REVIEW

Moore and Kommers (5) found that ordinary steels have a fatigue limit occurring about $10^6$ cycles of stress, but that in an 0.02 per cent carbon material the fatigue limit occurred at some $10^7$ cycles. In a similar vein, Lessells (9) showed an *S-N* curve for ingot iron which had not flattened out in $10^7$ cycles. Epremian and Mehl (10), found the knee of the *S-N* curve for an ingot iron at $6 \times 10^7$ cycles.

It is apparent from these tests that the position of the occurrence of the fatigue limit in iron-base alloys is dependent, at least in the low-carbon range, on the carbon content. If so, it might be expected that the yield point and associated strain aging phenomena are involved.

Moore and Alleman (11) were the first to report on the increased fatigue properties (finite life and fatigue limit) at elevated temperatures; a speed effect was also noted. They reported an increase of about 30 per cent in the fatigue limit in going from room temperature to 550 F (290 C). At higher temperatures the fatigue strength decreased again and no really flat fatigue limit was found.

Levy and Sinclair (12) have made an excellent study of strain aging effects in fatigue. They used specimens of SAE 1018 steel part of which were decarburized in a wet hydrogen atmosphere for four days at 1300 F (705 C), and part were treated in a helium atmosphere at the same temperature for 24 hr to produce an undecarburized material with an equivalent grain size and a comparable thermal history.

Their tests were made at various temperatures in rotating-beam machines at a constant cyclic speed of 2600 cpm and at an arbitrary stress of 35,000 psi. Their results on the helium-treated steel at various temperatures show a maximum fatigue life at about 460 F (240 C). This peaking temperature is in good agreement with that calculated on the basis of strain aging theory.

Tests on the hydrogen-treated material also showed a peak fatigue life; however, in specimens given a more thorough decarburization no such peak was found; the fatigue life shortened continuously up to 420 F (215 C). Thus, "as carbon and nitrogen were removed from solution there was a progressive decrease of the peaking tendency in fatigue life" (12).

Levy and Sinclair do not publish the carbon analyses for their heat-treated specimens and it is accordingly impossible to evaluate their data or to compare it with that reported below. However, an extrapolation of the present data would show that at 35,000 psi in a rotating-beam test our specimens should break in about $10^4$ cycles while their mean life at room temperature was $9.93 \times 10^4$ cycles. Thus, it would seem that the carbon and nitrogen content of the specimens here used is somewhat lower than those reported by Levy and Sinclair.

The present authors (13) noted what seemed to be typical non-ferrous behavior in a very-low-carbon iron, and it is on the basis of this observation and the work of Levy and Sinclair that the present work was undertaken.

While this work was in progress, Rally and Sinclair (14) reported on a further study of the strain aging phenomenon. They assumed that the presence of a knee in the *S-N* curves for steel is caused by strain aging which takes place during the fatigue test. They state that the position of the knee should thus depend on the amount of carbon and nitrogen in solution and, as well, on the time-temperature relation for the diffusion of these elements. The above working hypothesis predicts that the complete removal of carbon and nitrogen from the steel should result in the disappearance of the knee. Experimentally, of course, the complete removal of these elements is difficult to achieve. Rally and Sinclair suggest that the major effect of reducing carbon and nitrogen is to increase the mean distance over which the carbon must travel to achieve the effective pinning of dislocations which is the cause of strain aging. The net effect predicted, then, is a longer life (in cycles) at the knee because of the longer time needed by the carbon to travel the extra distance.

Their experimental work was done on an SAE 1018 steel which they decarburized in a wet (?) hydrogen atmosphere for two weeks at a temperature of 1300 F (705 C). They also treated samples of this steel in helium for 36 hr at 1300 F (705 C). The results of their room temperature tests show that the helium-treated but undecarburized steel exhibited a knee at $3.7 \times 10^6$ cycles and a mean fatigue limit stress level of 27,500 psi in a rotating cantilever test; the hydrogen treated steel showed a knee at $7.4 \times 10^6$ cycles and 24,080 psi; also by renitriding the steel after decarburization they were able to obtain a knee in the *S-N* curve at $3.2 \times 10^6$ cycles and 39,300 psi. Their other results on the helium-treated steel, in rotating-beam tests, show the knee position to shift to the left with temperature increase to 440 F (225 C), as would be expected due to the increased strain aging in this temperature range. They conclude that the removal of the elements associated with strain aging allows the knee of the *S-N* curve to shift to the right and that if these elements could be sufficiently removed so as to eliminate strain aging altogether the knee might be eliminated completely.

On several counts these results of Rally and Sinclair are disturbing. The published stress-strain curves for the original as-received cold-rolled stock and for the helium-treated specimens show that the helium-treated steel has an appreciably higher lower yield point although the helium-treated steel is dead soft and almost certainly of a larger grain size than the as-received stock. More disturbing is the fact that the finite life portions of the *S-N* curves for the helium-treated steel tested in fatigue at various temperatures show a maximum life at room temperature—in apparent contradiction to the results of Levy and Sinclair. (That the tests were run at different speeds—faster by Rally—should make only a small difference in the temperature of maximum life and should raise it.) The fatigue limits as a function of temperature (up to 440 F) do not show any large effect (27,000 ± 2000 psi will cover the range) nor any trend to higher values at higher temperatures nor the reverse. This is in opposition to the results of Moore and Alleman (11) on much the same steel.

TABLE I.—MECHANICAL PROPERTIES OF AS-RECEIVED STOCK[a] (0.252-IN. DIAMETER SPECIMENS).

| | |
|---|---|
| Upper Yield Point, psi | 38 670 |
| Lower Yield Point, psi | 37 460 |
| Ultimate Tensile Strength, psi | 58 400 |
| Reduction of Area, per cent | 71.9 |
| Elongation in 1-in. gage length, per cent | 36.0 |
| Rockwell Hardness, B scale | 62–64 |

[a] Average of five specimens.

NOTE.—All fractures were cup and cone type.

## EXPERIMENTAL WORK

The specimens used in this investigation were prepared from an SAE 1008 steel of the following composition in per cent:

| | |
|---|---|
| Carbon | 0.09 |
| Silicon | 0.14 |
| Manganese | 0.46 |
| Phosphorus | 0.010 |
| Sulfur | 0.027 |
| Chromium | 0.08 |
| Titanium | 0.01 |
| Molybdenum | 0.01 |
| Nickel | 0.07 |
| Copper | 0.05 |

The material was an exceptionally clean silicon killed-steel, having a cleanliness rating well above SAE O-O-A. This material (hereinafter referred to as "as-received" or "AR stock") was obtained as hot-rolled rod; microscopic examination revealed an equiaxed structure with a grain size of ASTM Nos. 7 to 9. The static tensile and hardness properties of the AR stock are given in Table I. The tension tests were performed on standard ASTM 0.252-in. diameter specimens having a 1-in. gage length.[4]

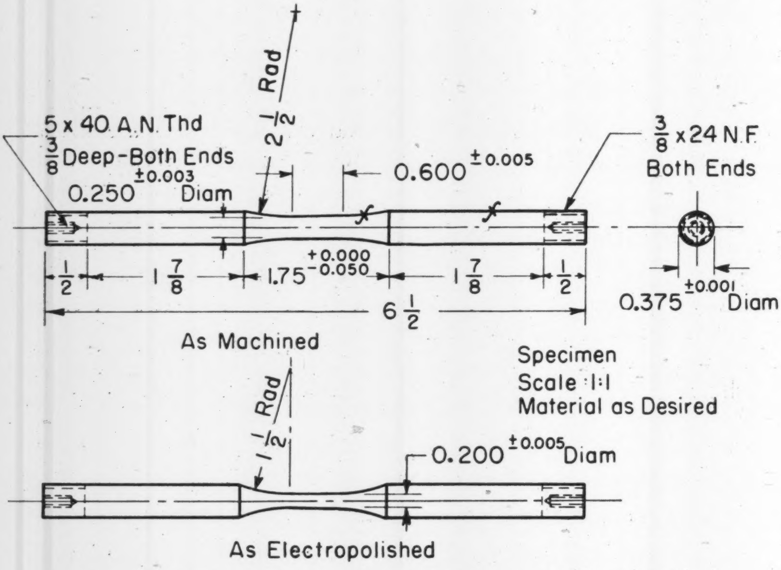
5 x 40 A.N. Thd
$\frac{3}{8}$ Deep-Both Ends
$0.250^{\pm 0.003}$ Diam
$2\frac{1}{2}$ Rad
$0.600^{\pm 0.005}$
$\frac{3}{8}$ x 24 N.F.
Both Ends
$\frac{1}{2}$
$1\frac{7}{8}$
$1.75^{+0.000}_{-0.050}$
$1\frac{7}{8}$
$\frac{1}{2}$
$6\frac{1}{2}$
$0.375^{\pm 0.001}$ Diam
As Machined
Specimen
Scale 1:1
Material as Desired
$1\frac{1}{2}$ Rad
$0.200^{\pm 0.005}$ Diam
As Electropolished

FIG. 1.—Specimen Design for Tension-Compression Fatigue Fixture.

The testing program of this research consisted of three parts. The first was the determination of the *S-N* curve for the AR material in axial tension-compression using the "staircase method" (**15**) for determining the fatigue limit. Twenty-six specimens were used for this purpose.

The second portion of the work was the *S-N* curve determination for the decarburized material in axial tension-compression. Seventeen of these specimens were tested. The exceptionally long lives of the specimens made the testing at the slow speed of the tension-compression machine costly; for this reason further work on the decarburized mate-

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[4] Tentative Methods of Tension Testing of Metallic Materials (E 8 – 57 T), 1957 Supplement to Book of ASTM Standards, Parts 1 and 2.

TABLE II.—MECHANICAL PROPERTIES OF AS-DECARBURIZED STOCK[a] (0.252-IN. DIAMETER SPECIMENS).

| | |
|---|---|
| Lower Yield Point, psi | 12 090 |
| Ultimate Tensile Strength, psi | 40 880 |
| Reduction of Area, per cent | 85 |
| Elongation in 1-in. gage length, per cent | 47.5 |
| Rockwell Hardness, E scale | 67–70 |

[a] Average of five specimens.

NOTE.—All fractures were cup and cone type.

rial was carried out on R. R. Moore rotating-beam machines. Twenty-six specimens were so tested.

All the tension-compression tests herein reported were completely reversed tests, that is, all tests were made about zero mean stress.

Tension-compression test specimens, shown in Fig. 1, were machined from the AR stock; some of them were decarburized and some were reserved for testing as received. The decarburization was accomplished in water-saturated hydrogen at 140 F (60 C) (16) in two stages: the first, 100 hr at 1725 F (940 C), and the second a further 100 hr at 1290 F (700 C); a slow overnight cooling from 1725 F to 1290 F produced a ferritic grain size of ASTM Nos. 3–5, which was not affected by the further decarburization. This material was then said to be in the "as-decarburized" condition (hereinafter referred to as the AD material). This treatment yielded specimens with 0.003 to 0.005 per cent carbon,[5] 0.009 per cent oxygen, 0.0003 per cent hydrogen and 0.00018 per cent nitrogen; the analysis of the other elements present is unchanged. After a few days at room temperature the specimens analyzed showed 0.00000 per cent hydrogen. The static mechanical properties of this material are given in Table II.

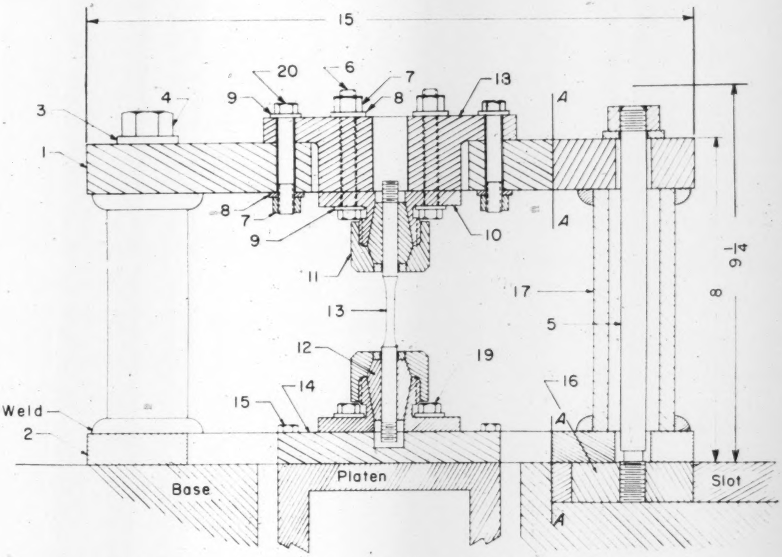

FIG. 2.—Front Section of the Assembled Testing Fixture.

The AR specimens were not heat treated in any fashion but all tension-compression (AR and AD) specimens were hand finished on a high-speed lathe through 0000 paper and then given a final electropolish to remove (about 0.005 in.) the polishing scratches and the cold-worked surface layers.

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[5] All low carbon analyses were performed at the Research Laboratory of the Allegheny Ludlum Steel Corp. through the courtesy of A. J. Lena.

The R. R. Moore specimens of the decarburized stock (hereinafter referred to as RD specimens) used in this investigation were the ASTM standard unnotched type specimen with a final radius of curvature of 5 in.

The RD specimens were rough machined to 0.250 in. minimum diameter with slightly oversized ends. Prior to decarburization the internal threads were plugged and the cone ends and end plugs were plated with about 0.003 in. of copper to inhibit decarburization of these sections. Since these specimens have the same minimum diameter as the tension-compression type specimens the same decarburization treatment was used and the resulting structure was the same. Following decarburization the specimens were finish machined and longitudinally ground in accordance with ASTM specifications to about 0.225 in. in diameter, and then the gage section was electropolished; the final minimum diameter of these specimens was about 0.220 in.

The tension-compression specimens were tested in a Sonntag SF-1-U Universal Fatigue Machine, operating at 1800 cpm. For these tests, the specimen was fixed vertically between the moving platen and a stationary jig by friction collets at both ends. A section through the fixture is shown in Fig. 2.

The details on the construction of the fixture and the alignment procedure have been published elsewhere (8); for these tests the alignment procedure was carried (by trial and error) to an apparent eccentricity of less than 0.0005 in.—the maximum longitudinal stress in the specimens as rigidly mounted was about 340 psi.

*Testing Procedure, Errors and Calculations:*

For testing the AR, AD, and RD specimens the criterion used was the peak nominal stress of the cycle. The minimum diameter of each specimen was measured optically to 0.0001 in. on a comparator, at a magnification of 20×, the cross-sectional area was calculated and the applied stress found using the following formulae:
For AR and AD specimens:

$$S = \frac{F}{A}$$

where:

$S$ = applied stress, psi,
$F$ = applied load, lb, and
$A$ = cross-sectional area, sq in.

For RD specimens:

$$S = \frac{Mc}{I}$$

where:

$S$ = stress at the surface fibers, psi,
$M$ = applied bending moment, in.-lb, and
$\frac{I}{c}$ = section modulus at the minimum section, in.$^3$

The maximum percentage error in the applied stress was about ±0.7 per cent for both types of tests. In addition, for the tension-compression tests on the AR and AD specimens the maximum stress introduced into the surface fibers of these specimens through misalignment of the test fixture was about ±340 psi or about ±3 per cent of the applied stress. Finally, the maximum shear stress introduced into these specimens due to twisting of the collet in its chuck was experimentally determined as about 450 psi.

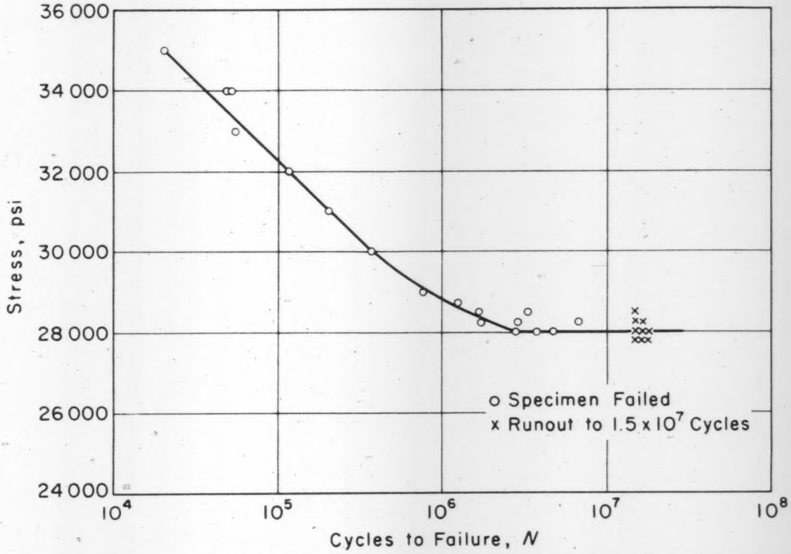

FIG. 3.—Fatigue Behavior Plot for Polycrystalline As-Received Specimens; Tested in Tension-Compression.

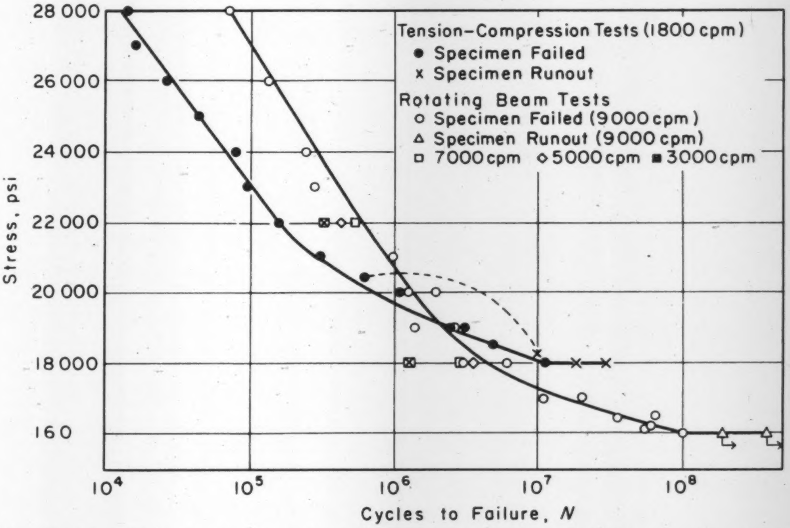

FIG. 4.—Fatigue Plots for As-Decarburized Specimens in Tension-Compression and Rotating Beam Tests.

## EXPERIMENTAL RESULTS AND DISCUSSION

The fatigue properties of the AR specimens tested in completely reversed axial tension-compression are shown in Fig. 3. The fatigue limit knee occurs at about $2.5 \times 10^6$ cycles. The fatigue limit, roughly determined by the staircase technique (**15**), is about 28,000 psi. The fatigue behavior of this material is not of great significance *per se*, but these data are useful for comparison purposes to show the effect of the removal of carbon and nitrogen from this steel. (There is, of course, the aforementioned accompanying change in grain size.) It may be noted that these results are very close to those of Rally and Sinclair (**14**) on SAE 1018.

The results of tests on AD specimens as presented in Fig. 4 are for completely reversed axial tension-compression. These are incomplete results in that it was impractical to allow the unbroken specimens to run a sufficient time to be properly classed as runouts. Thus the position of the knee at about $10^7$ cycles and 18,000 psi is not unequivocal. Data on ingot iron (**10**) show a fatigue limit appearing at from $5 \times 10^7$ to $6 \times 10^7$ cycles; these data also show a very wide scatter at stresses approaching the endurance limit.

One specimen, AD-16, tested at 18,250 psi, was stopped after $10^7$ cycles, heated to 1300 F (705 C) for 1 hr in dry hydrogen and furnace cooled; it was then reloaded to 20,400 psi and failed very close to the original curve. Taken at face value, this would indicate the presence of no cracks after $10^7$ cycles since a crack is expected to be the only form of fatigue damage not healable by annealing. However, this one result is not unequivocal but only an indication that a fatigue limit exists in the neighborhood of 18,000 psi for this material tested in axial tension-compression.

As indicated above, the work was continued in rotating-beam tests. The results of these last experiments are presented in Fig. 4. In these tests, the knee of the *S-N* curve is found at about $10^8$ cycles and 16,000 psi. The rotating-beam tests were run at 9000 cpm except for six tests that were made to check the effect of change in testing speed. It was found that a decreased life resulted when the testing speed was decreased to 3000 cpm.

It is to be noted that all tests were run at stresses above the yield point. In the axial tension-compression tests the test stresses (neglecting experimental errors in load and area determinations) are known accurately; in the R. R. Moore tests the surface stresses are in reality unknown because the stress distribution is not linear as was assumed. However, there are no points common to the two curves at which comparisons can apparently legitimately be made. The usual assumption that the stresses at the fatigue limit, or at a given life, are in fact equal, leads to the unorthodox conclusion that the actual stress in an R. R. Moore test run above yielding is higher than that calculated from supposed elastic conditions. The situation is confused by other factors as well: there is the possible size effect (more material is at peak stress in the axial tests), but this is almost certainly negligible; there is the possible stress gradient effect, of no known effect *per se*; and there is a possible speed effect in the stress value of the fatigue limit as well as on the finite life.

Whether or not the surface stresses in an R. R. Moore test run above the yield point are known or not, it is certain that the relative stresses increase with increasing nominal stress. And it has been demonstrated many times (**17**) that specimens tested above the yield strength exhibit "elastic" properties (very small hysteresis) after the first few cycles.

The results reported here and the work of others suggest that the presence of a fatigue limit is due to the two competing processes of normal fatigue damage accumulating with cycles, tending toward failure but opposed by the strengthening effect of strain aging in plastically deformed material. Below the fatigue limit, strain aging predominates and fracture does not follow. Above the fatigue limit, damage accumulates faster than strain aging can offset and fracture does follow. The sharp knee of the *S-N* curve is the transition. Below the fatigue limit, the strain hardening and strain aging strengthening effects are postulated to predominate; therefore, understressing and coaxing effects are to be expected. In a sense, then, the fatigue limit knee is not the start of a horizontal line but rather the start of a line which, at least initially, slopes upward to higher stresses at longer lives. However, since the *S-N* diagram as normally drawn is restricted to constant stress tests, this concept is not strict.

Some of the pieces of such a picture are published. Levy and Sinclair (**12**) have shown that strain aging does in fact occur under fatigue loading. The present authors and Rally and Sinclair (**14**) have shown the slower appearance, at longer life, of a fatigue limit with lowered carbon content. Several pieces, however, remain obscure.

It is not immediately apparent what strain aging does to counteract fatigue damage. Does it prevent cracking or merely inhibit strongly the growth of a fatigue crack? The observation, at least in notched specimens, that fatigue cracks need not propagate continuously, suggests that the effect is to strengthen the lattice ahead of the advancing crack so that propagation becomes impossible. The results reported by Frost (**18**), can be interpreted in this fashion as it was observed that a fatigue crack starting from a notch propagated only to the point where the stress, calculated from the original stress concentration at the notch, was just equal to the fatigue limit. There may also be an effect on crack initiation. Since the number of cycles to effect crack initiation is relatively small, at least in soft, ductile materials, it is not thought that this will be a large effect as measured against total life in the finite life region.

But accepting the effect of strain aging as that due to inhibiting propagation does not immediately simplify the problem. Since it is expected that the effective strain aging will be restricted to the local area ahead of the crack where plastic deformation is high, the diffusion rates cannot be assumed to be those operative in large specimens under little stress. It has been shown (**19**) that diffusion is faster under fatigue loading than under no stress, and even this experimental result is an average over a large amount of unaffected and a small amount of affected material. This can most easily be interpreted to be a result of the local rise in temperature necessarily accompanying the passage of dislocations in plastic flow (**20**). Against this is the agreement between theory and the experiments of Levy and Sinclair using diffusion data obtained in annealed materials. The agreement can be considered somewhat fortuitous.

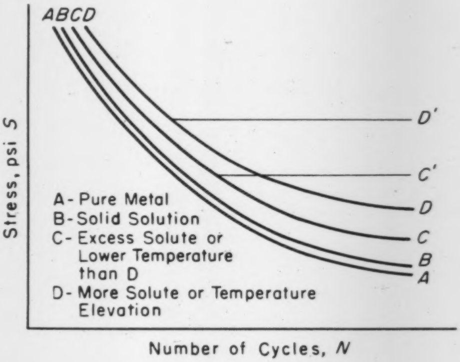

FIG. 5.—Schematic Fatigue Diagram.

Neglecting the last point, however, one can attempt to be more specific on the details of the process. It is assumed that the *S-N* curve for pure iron is a smooth function of the applied stress (a consideration believed to be applicable to all pure metals) and that the peculiar slip behavior of iron is not effective in determining fatigue behavior of the pure metal (**8**). Curve *A* of Fig. 5 is a schematic representation of this behavior.

The addition of carbon (or nitrogen)

to iron must be considered to cause several separate effects. There is the solid solution strengthening qualitatively analagous to that caused by any solute; if excess carbon (or nitrogen) is present the carbide phase will cause additional strengthening. These strengthening effects lead to longer life at a given stress under fatigue conditions, and the *S-N* curve is shifted to the right. These effects are shown in curve *B* of Fig. 5.

In addition, the presence of carbon (or nitrogen) conveys the power to strain age—with time a strengthening occurs greater than that due to strain hardening alone. This also leads to a longer life at a given stress, with the increase in life becoming more prominent with decreasing stress. The result is a further shift of the *S-N* curve—this time upward and to the right as shown by the smooth curve *C*, Fig. 5.

Strain aging with the carbon (or nitrogen) content, temperature, and speed of testing constant, is not expected to be the strong function of the stress level that the damage occurring in a single reversal of stress obviously is. Independent of the strengthening due to strain hardening, then, it is to be expected that there will be some limiting lower stress at which the strengthening due to strain aging will be great enough to offset the weakening due to the deformation; this stress, shown by curve *C′*, Fig. 5, is the fatigue limit.

Increasing the carbon (or nitrogen) content will increase the fatigue limit stress because the amount of strain aging, in terms of the breakaway stress (**22**), is expected to be a relatively strong function of the carbon (or nitrogen) content. This effect is reflected in curve *D-D′*, Fig. 5. Since the shift of the fatigue limit to higher stresses effectively shifts the knee to the left, the two effects of strain aging are in opposition and it is not obvious whether the knee will shift to the right or left. It is only the accident that the second mentioned effect on the knee predominates that makes the over-all effect on the fatigue limit knee a shift to shorter life with increasing carbon (or nitrogen) content. It is to be expected that the strain aging as measured by the maximum possible strengthening will be a strong function of carbon (or nitrogen) content and that the effect of speed of testing and temperature will be on the realized percentage of this maximum possible strengthening at a given carbon (or nitrogen) content.

There is then an expected effect of carbon (or nitrogen) content in terms of the longer distances of diffusion necessary to achieve effective pinning of dislocations. This effect should be manifest as a speed (or temperature) effect; that is, for a given carbon (or nitrogen) content, the percentage of maximum possible strain aging which results will increase with slower testing speed at a constant temperature. The speed effect measured here, on the basis of admittedly too few data, is apparently in the wrong direction.

This picture of the role of strain aging in fatigue of irons and steels differs from that of Rally and Sinclair (**14**). The complete removal of carbon (or nitrogen) from iron will result in the disappearance of the fatigue limit on both interpretations. Both interpretations suggest that the phenomenon of the fatigue limit is not a fatigue phenomenon, *per se*, and that the prototype of fatigue behavior is not steel but aluminum or copper.

But the effects predicted differ considerably when considering the behavior of iron containing small amounts of carbon (or nitrogen). Rally and Sinclair predict the shift of the knee of the *S-N* curve to the right because of the depletion of the carbon (or nitrogen) in solution and the necessarily longer distance that the interstitial atoms must travel to achieve effective pinning of dislocations. The present explanation suggests that the major effect of the removal of carbon (or nitrogen) is to lower the fatigue limit—because the *S-N* curve sloped downward to the right, the knee makes its appearance at longer life. There is also little question that the amounts of carbon (or nitrogen) available are much greater than the solubility limit; the same may not be true of nitrogen.

The strain aging must be as complete as it will ever be within a few cycles of the deformation, perhaps not in the single cycle assumed by Levy and Sinclair, but certainly in very few. Strain aging must essentially maintain pace with the damage occurring from the deformation, independent of whether it affects crack initiation or crack propagation. It cannot be expected to catch up after $10^5$ or $10^6$ cycles.

The present interpretation, then, is compatible with the results of Levy and Sinclair; the strengthening due to more effective strain aging, achieved by increasing the temperature to about 460 F (240 C) must lead to longer life at a given stress (see Fig. 5). The weakening due to less effective strain aging, achieved by lowering the carbon (or nitrogen) content in the present work, can lead to shorter life as it does; here the issue is less clear cut because of the loss of strength due to decreased solution hardening. There is a surprising lack of loss of strength in the finite life region between the helium-treated and hydrogen-treated specimens of Rally and Sinclair. There is the more expected result of an increase in life reported by Moore and Alleman (**11**).

The present interpretation agrees with the results of Moore and Alleman who found a large increase in the fatigue limit on increased testing temperature. The strengthening due to more effective strain aging raises the stress level at which damage can be repaired by strain aging. Again there is the surprising lack of effect reported by Rally and Sinclair. The situation is not clear and further work is necessary and in progress.

It is to be noted that the strain aging envisioned here may or may not be strong enough to cause strain aging after tensile straining. The deformation to cause a fatigue crack must be intense but very local, and general strain aging may not occur. In addition, virtually all steels show strain aging effects in a notch-impact test. Fatigue tests may be as sensitive to local strain aging, if not more sensitive, than are the notch-impact tests. It is apparent that the same interpretation applies equally well to the case of molybdenum and titanium—materials which contain interstitial impurities. Whether the fatigue limit in austenitic stainless steel is due to some aging or to a transformation phenomenon (perhaps martensitic) cannot at present be answered.

The complete substantiation of the interpretation common to this work and that of Rally and Sinclair, that the fatigue limit is not a fatigue phenomenon but rather an "extraneous" effect of strain aging, would not appear to be possible with the iron-carbon system. The uncertainties attendant to extrapolation would seem to insure doubt. However, if the introduction of an alloying element that will cause strain aging in a typical non-ferrous material can be shown to produce a fatigue limit in that material, the substantiation of the present hypothesis would be nearly complete. Metallographic and X-ray evidence showing that the fatigue limit in austenitic materials is due to some precipitation or transformation phenomenon (perhaps martensitic) would essentially complete the picture.

## CONCLUSIONS

1. The partial removal of carbon and nitrogen from an SAE 1008 steel results in lowering the life at a given stress and a lowering of the fatigue limit; the concurrent result is that the fatigue limit knee occurs at longer life. The known effects of strain aging will account for this, which is probably the only feasible explanation.

2. The results suggest that the complete removal of carbon and nitrogen will suppress the fatigue limit, if it exists at all, to lifetimes beyond practicable tests, approaching non-ferrous behavior.

3. It can be inferred that the fatigue limit is not directly a consequence of the cyclic stressing but its appearance is due to the occurrence of strain aging during testing.

4. There is a larger difference between axial tension-compression tests and R. R. Moore tests on the decarburized material than might be expected.

5. There is a larger speed effect than might be expected; this is intimately related to the strain aging in this material.

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## REFERENCES

(1) H. James and D. Galton, "Report of the Commissioners Appointed to Inquire into the Application of Iron to Railway Structures," Wm. Clowes and Sons, London, Her Majesty's Stationery Office, July 26, 1849.

(2) W. Fairbairn, "Experiments to Determine the Effect of Impact, Vibratory Action and Long-Continued Changes of Load on Wrought Iron Girders," *Philosophical Transactions*, Royal Society, London, Section A, Vol. 154, Part 1, p. 311 (1864.)

(3) A. Wöhler, "Versuche über Verdrehhung von Eisenbahnwagon-Achsen während der Fahrt," *Zeitunc fur Bauwesen*, Vol. 8, p. 642 (1858). For other papers see also Vol. 10, p. 583 (1860); Vol. 13, p. 233 (1863); Vol. 16, p. 67 (1866); and Vol. 20, p. 73 (1870). This work is abstracted in *Engineering*, London, Vol. 11, pp. 199, 221, 243, 299, 326, 349, 397, and 439 (1871).

(4) J. Bauschinger, "Über die Veränderung der Elasticitätgrenze und Festigkeit de Eisens," *Mitteilunger Mechanischer Technischen Laboratories*, Konigliche technisihe Hochschule, München, (1886).

(5) H. F. Moore and J. B. Kommers, "An Investigation of the Fatigue of Metals," University of Illinois Engineering Station Bulletin No. 124 (1921).

(6) H. F. Moore and T. M. Jasper, "An Investigation of the Fatigue of Metals," University of Illinois Engineering Station Bulletin No. 142 (1924).

(7) C. S. Barrett, "The Structure of Metals," McGraw-Hill Book Co., New York, N. Y., 2nd Edition, p. 340, (1952).

(8) H. A. Lipsitt and G. T. Horne, "The Behavior of Single Crystals of Iron Under Fatigue Loading," International Conference on Fatigue of Metals, p. 513 (1956), The Institution of Mechanical Engrs. (London).

(9) J. M. Lessells, "Discussion to the Report of the Research Committee on Fatigue of Metals," *Proceedings*, Am. Soc. Testing Mats, Vol. 41, p. 151 (1941).

(10) E. Epremian and R. F. Mehl, "Investigation of the Statistical Nature of Fatigue Properties," *NACA Technical Note 2719*, June 1952.

(11) H. F. Moore and N. J. Alleman, "Progress Report on Fatigue Tests of Low Carbon Steel at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 114, (1931).

(12) J. C. Levy and G. M. Sinclair, "An Investigation of Strain Aging in Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 866, (1955).

(13) H. A. Lipsitt and G. T. Horne, "Investigation of the Fundamentals of Fatigue in Metals," Final Report to NACA, Research Contract NAw-6331, January 15, 1955.

(14) F. C. Rally and G. M. Sinclair, "Influence of Strain Aging on the Shape of the *S-N* Diagram," *Report No. 87*, Dept. of Theoretical and Applied Mechanics, University of Illinois, June 1955.

(15) J. T. Ransom and R. F. Mehl, "The Statistical Nature of the Endurance Limit," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 185, p. 364 (1949).

(16) H. Schenck, Translated by H. J. Goldschmidt, *Physical Chemistry of Steelmaking*, British Iron and Steel Research Assn. p. 167, (1945).

(17) H. J. Gough, "The Elastic Limits of Copper Under Cyclical Stress Variations," *Engineering*, Vol. 114, p. 291 (1922); G. E. Dieter, R. F. Mehl, and G. T. Horne, "The Statistical Fatigue Properties of Lamellar and Spheroidal Eutectoid Steel," *Transactions*, Am. Soc. Metals, Vol. 47, p. 423 (1955).

(18) N. E. Frost, "Crack Formation and Stress Concentration Effects in Direct Stress Fatigue," *Engineer*, Vol. 200, p. 464 and 501 (1955).

(19) H. Schenck and E. Schmidtmann, "Effect of Alternating Stress on Diffusion and Segregation Processes in Unalloyed Steels," *Archiv für das Eisenhuttenwesen*, Vol. 25, p. 579 (1954).

(20) A. M. Freudenthal and J. H. Weiner, "On the Thermal Aspect of Fatigue," *Journal of Applied Physics*, Vol. 27(I), p. 44 (1956); J. D. Eshelby and P. L. Pratt, "Note on the Heating Effect of Moving Dislocations," *Acta Metallurgica*, Vol. 4, No. 5, Sept., 1956, p. 560.

(21) C. Wert, "Precipitation out of Dual Solid Solutions of Carbon and Nitrogen in Alpha-Iron," *Acta Metallurgica*, Vol. 2, No. 3, May 1954, p. 361.

(22) A. H. Cottrell, "Dislocations and Plastic Flow in Crystals," Clarendon Press, Oxford, England, p. 143 (1953).

# DISCUSSION

Mr. Davis S. Fields, Jr.[1]—If strain aging is an influencing factor in fatigue, might we not expect a dependence of fatigue results upon the frequency of the test? Also might there be an effect of interrupting a fatigue test and letting the specimen rest for a while?

Mr. H. A. Lipsitt (*author*).—Replying to Mr. Fields, Levy and Sinclair[2] have postulated the frequency and temperature dependence of strain aging in fatigue. This dependence is such that a higher frequency or lower temperature results in achieving only a portion of the maximum possible strain aging. This behavior is to be expected since diffusion is the controlling factor in the strain aging process.

In answer to the second question, Fulmer Research Inst. has recently completed some work along this line for the United States Air Force under Contract AF 61(514)-890. The results indicated only a very minor increase (20 per cent) in fatigue life for ingot iron alternately fatigued and aged (at 930 F for 15 min) until failure occurred. The same sort of result would be anticipated for room temperature aging.

Tests on alpha brass (70 per cent copper—30 per cent zinc) were also performed using rest periods at room and elevated temperatures. This series of tests showed no significant recovery effect on fatigue life. Sinclair and Dolan had previously reported similar results on alpha brass in the *Proceedings* of the First National Congress of Applied Mechanics.

Mr. H. T. Corten.[2]—In commenting on one of the discussions you mentioned that alpha brass was studied in terms of rest periods and the influence upon fatigue life. Did you expect to find strain aging and therefore any influence on the fatigue life in alpha brass?

Mr. Lipsitt.—No effect on fatigue life was anticipated for the alpha brass specimens described above; rather this work was done so that a comparison could be made with the results achieved using ingot iron. It is also interesting to note that a yield point has been observed in alpha brass at about 200 C and thus, according to our postulates and those of Levy and Sinclair, it is entirely possible that a strain aging effect could be observed in the fatigue of alpha brass at the proper combination of cyclic frequency and temperature.

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[2] See reference (**12**) of paper.

[2] Research Associate Professor of Theoretical and Applied Mechanics, Talbot Laboratory, University of Illinois, Urbana, Ill.

# EFFECT OF GRINDING CONDITIONS AND RESULTANT RESIDUAL STRESSES ON THE FATIGUE STRENGTH OF HARDENED STEEL*

By L. P. Tarasov,[1] W. S. Hyler,[2] and H. R. Letner[3]

## Synopsis

The fatigue limit of a hardened steel was investigated as a function of the residual stresses generated in flat test bars by longitudinal surface grinding, the experimental variables being wheel hardness, grinding fluid, and unit downfeed. Reversed bending fatigue tests showed that for good commercial grinding conditions the fatigue limit was the same as that obtained under gentle grinding. Severe grinding caused a decrease in fatigue limit of about 13 per cent, whereas some grinding with straight grinding oils resulted in increases up to about 38 per cent over the base-line fatigue limit.

The data indicated that there was an appreciable range in residual peak tensile stress (up to about 50,000 psi) for which the fatigue limit remained essentially at a base-line value. Above this residual tensile stress, reduction in fatigue limit occurred. This peak tensile stress occurred slightly below the surface. The surface stress was either tensile or compressive, depending upon grinding conditions. The marked increase in fatigue limit apparently resulted from a combination of high residual compressive stress and mechanical deformation at the surface.

Cyclic stressing did not reduce the magnitude of the residual stresses.

The scatter in the data appeared to be associated with metallurgical defects and with the normal variation in magnitude of the residual stresses obtained during grinding.

A previous paper by Tarasov and Grover (1)[4] presented a preliminary evaluation of the effects of grinding, polishing, shot peening, and abrasive tumbling on the fatigue strength of a hardened ball-bearing type steel. The investigation indicated that careful grinding parallel to the length of the test bar did not have an adverse effect on the fatigue strength, but that severe grinding conditions decreased the fatigue strength by as much as 25 per cent. Mechanical working of the surface by such techniques as shot peening and abrasive tumbling resulted in a marked increase in fatigue strength. The effects observed were tentatively ascribed to the production of favorable or unfavorable residual stress patterns by these various processes, but no attempt was made at that time to measure such stresses.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 618.

Since that time, a technique suitable for evaluating grinding stresses in flat fatigue test bars was developed by Letner (2). Accordingly, when the present study was undertaken, involving a much wider variety of grinding conditions, it was considered desirable to include residual-stress measurements so as to determine the effect of grinding variables on the formation of residual stresses. A knowledge of the latter would show the extent to which they might affect the fatigue properties. In addition, the effect of cyclic stressing upon residual stresses was investigated.

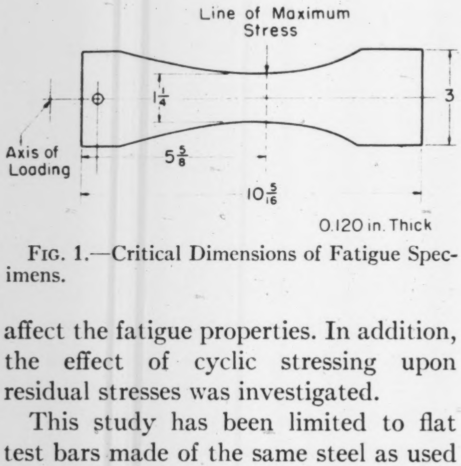

FIG. 1.—Critical Dimensions of Fatigue Specimens.

This study has been limited to flat test bars made of the same steel as used previously (modified AISI 52100 type), heat treated to Rockwell hardness C 59. Combinations of the grinding variables (wheel grade, unit downfeed, and grinding fluid) were chosen to include commercial grinding conditions as well as more extreme ones. This paper deals with the results obtained with longitudinally ground bars. The other part of this work, dealing with the effects of transverse grinding and of abrasive tumbling, will be discussed in a subsequent paper.

### *Acknowledgments:*

The entire investigation was planned and carried out as a three-way effort between Norton Co., Mellon Inst. of Industrial Research, and Battelle Memorial Inst. The test bars were prepared by Norton Co. The residual stress studies were carried out at Mellon Inst. as part of a wider investigation sponsored by the Grinding Wheel Inst. The fatigue tests and the metallographic and microhardness studies were carried out at Battelle Memorial Inst. under Norton Co. sponsorship.

The authors wish to acknowledge the help of Mr. R. J. Favor of Battelle Memorial Inst. in carrying out much of the experimental fatigue program and in the preparation of this paper and of Mr. R. D. Buchheit for the metallographic and microhardness studies.

## SPECIMEN PREPARATION

### *Preliminary Processing:*

The analysis of the steel, shown below, was practically the same as in the previous study (1). The steel was rated satisfactory by the supplier with respect to inclusions and cleanliness from the standpoint of use as a ball-bearing steel.

| Composition | Per Cent |
|---|---|
| Carbon | 1.01 |
| Silicon | 0.36 |
| Manganese | 0.27 |
| Sulfur | 0.011 |
| Phosphorus | 0.011 |
| Chromium | 1.25 |
| Vanadium | 0.19 |
| Iron | Balance |

Rectangular blanks were sheared slightly oversize from 0.200-in. thick hot-rolled and mill-annealed plate, the length being parallel to the rolling direction. The edges of the blanks were machined to 3 by $10\frac{5}{16}$ in. in section and the elliptical contours were sawed out oversize in preparation for subsequent grinding to the shape shown in Fig. 1. The bars were then ground wet to a thickness of 0.160 in. on a Blanchard surface grinder, with approximately equal amounts of stock being removed from both sides.

After the bars had been annealed 4 hr at 1100 F between heavy steel plates which were bolted together to preserve flatness, the bars were individually austenitized 15 min in a salt bath at 1550 F, oil quenched, and tempered 2 hr at 450 F to obtain a uniform Rockwell hardness of C 59. Some of the test bars were found to be warped more than was considered permissible in view of the amount of stock that had to be left available for the subsequent grinding operations. These bars were again annealed between the steel plates, this time for 2 hr at 1300 F. They were then hardened as before. The resulting microstructure of the finished test bars was a uniform dispersion of fine carbide particles in tempered martensite. Any surface decarburization was removed in the early stages of the subsequent grinding operations.

The next step was to grind the hardened bars as flat as possible to a thickness of 0.140 in. Approximately equal amounts of stock were removed from both sides, except for the differences induced by the slight deviations from flatness that resulted from the heat treatment. Grinding was done wet with a soft, porous-type wheel (32A46-F12VBEP), 10 in. in diameter and 3 in. wide, on a horizontal-spindle reciprocating-table surface grinder. The bars were ground carefully in order that the residually stressed layer would be shallow enough to be removed completely in the early stages of the final grinding operation. The unit downfeed was 0.001 in. except for the final 0.002 in. where a unit downfeed of 0.0005 in. was used. The grinding fluid was a general purpose soluble oil used at a 2 per cent concentration in water. This operation was performed in the machine shop and no record is available of the other grinding conditions, but it is known that they were characteristic of good commercial grinding practice.

The elliptical contours were ground with a soft wheel, generally 32A46-H8VBE, in the manner previously described (1) except that wet grinding with a 1 per cent water solution of a commercial nitrite-amine rust inhibitor was substituted for the dry grinding, in order to increase the rate of production without overheating the surface. The grinding fluid had to be fed through the wheel by means of a specially designed collet since it did not reach the area of grinding contact when applied conventionally through an external nozzle.

### *Final Grinding:*

After preliminary processing, the specimens were ground under various conditions to a final thickness of 0.120 in. at which point they were ready for the determination of grinding stresses and fatigue properties. In general, 16 bars were prepared in each group, of which 15 were used in the fatigue studies and the other one in the residual stress study.

The final grinding to 0.120 in. was done on the same horizontal-spindle, reciprocating-table surface grinder used in the previous study. The test bars were ground lengthwise individually, with a total of 0.010 in. being removed from each side. The wheel was diamond dressed with a moderately fast crossfeed to leave the wheel face open. This was done before each side was ground, so that the condition of the wheel face would be duplicated as nearly as possible during the last downfeed on both sides of all the test bars comprising a group.

The grinding conditions kept constant throughout this study were the same as in the earlier work and were as follows: wheel speed, 6000 surface ft per min; wheel size, 8 to 7 in. in diameter, $\frac{3}{4}$ in. wide; table speed, 60 to 65 ft per min; and unit crossfeed, 0.050 in. after each table traverse.

The grinding variables included the wheel grade, the unit downfeed, and the grinding fluid. Three wheel specifications were used, differing only in the grade: 38A46 - H8VBE, 38A46 - I8VBE, and 38A46-M8VBE. These are white, vitrified bond, friable aluminum oxide wheels, such as are commonly used for surface grinding hardened steels. The I grade is the type that normally would be recommended for grinding the test bars with soluble oils. The H grade is softer and was included because it had been used in previous work. The M grade, which is much too hard for this type of grinding, especially with soluble oils, was used in order to have some extremely severe conditions conducive to the generation of high tensile stresses, such as would be expected to lower the fatigue properties appreciably. The unit downfeeds following each completed crossfeed were 0.0001, 0.001, or 0.002 in.

TABLE I.—SUMMARY OF GRINDING VARIABLES.

| Test Group[a] | Wheel Grade | Unit Downfeed, in. | Grinding Fluid |
|---|---|---|---|
| AA..... | H | $0.1 \times 10^{-3}$ | Soluble oil B |
| AB..... | H | 0.1 | Soluble oil B |
| AC..... | M | 2.0 | Soluble oil B |
| AD.... | M | 1.0 | Grinding oil I |
| AE..... | M | 2.0 | Grinding oil I |
| AF..... | I | 1.0 | Grinding oil I |
| AG.... | I | 1.0 | Grinding oil L |
| BA..... | I | 1.0 | Grinding oil L |
| BC..... | I | 1.0 | Soluble oil F |

[a] Test groups starting with A were hardened once, those with B, twice. AG and BA furnish a comparison of the two heat treatments. The surface grinder was overhauled completely in the interval between the preparation of AA and AB. In order to speed up the preparation of the AA and AB bars, heavier downfeeds were used in the earlier stages of grinding, these being gradually decreased so that the six final downfeeds were 0.0001 in. each.

The fluids included two soluble oils (designated B and F) and two sulfochlorinated grinding oils (designated I and L). Dry grinding was used in the earlier study but not in the present one because Letner (3) has established that, for the conditions of this investigation, the residual grinding stresses are essentially the same for dry grinding as for grinding with a variety of soluble oils.

Soluble oils B and F and grinding oil I were among those studied by Letner, whose code designations are used in this paper. Soluble oil B had a nominal composition of 18 per cent mineral oil, 25 per cent surface-active compounds including emulsifiers and soaps, 5 per cent other organic materials, and 52 per cent water. The corresponding figures for soluble oil F are 70 per cent, 24 per cent, 3 per cent, and 3 per cent. Water emulsions containing 2.5 per cent and 1.25 per cent, respectively, of the concentrated product were used for grinding.

Grinding oil I is a sulfochlorinated mineral oil with a very high content of sulfochlorinated fats; the total sulfur content was about 3.5 per cent. This oil, commonly used for thread grinding, is a very dark oil, high in active sulfur, with a Saybolt universal viscosity of 190 sec at 100 F. Grinding oil L is also a sulfochlorinated mineral oil, having a viscosity of 230 sec and with about the same sulfur content but only a moderate amount of fat. However, the sulfur is compounded differently in oil L so that it is transparent and nonstaining. It becomes more active than oil I at the point of grinding contact, as shown by other grinding tests. Oil L was used as a check upon oil I, which resulted in a few microscopic corrosion pits that might have had a detrimental effect upon the fatigue properties of the finished test bars.

The combinations of these variables for which stress and fatigue data were obtained are listed in Table I.

## RESIDUAL STRESS STUDY

### *Description of Method:*

Residual stresses were determined by the deflection method, using a comparator based on the differential-transformer principle (2). Changes in curvature parallel and perpendicular to the grinding direction (the principal stress directions) were measured as successive uniform layers were etched from the surface of a square specimen taken from a test bar. The etchant was a 10 per cent solution of nitric acid in either alcohol or water.

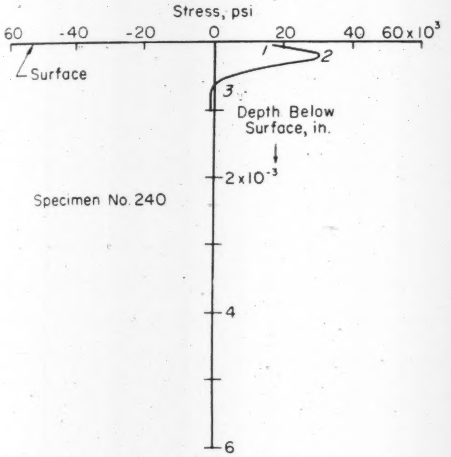

FIG. 2.—Typical Stress Distribution in Group AA Specimens.

Although the stresses were determined both parallel and perpendicular to the grinding direction, only the former are considered in this paper. The stress distributions in these two directions were found to be very nearly the same, the principal difference being that the perpendicular stresses were always somewhat lower algebraically than the parallel ones. Thus, the perpendicular tensile stresses were smaller and the compressive stresses larger but they could be neglected since the maximum tensile stress appeared to be the controlling factor with respect to fatigue properties. The stresses were calculated as those present in a specimen which is constrained to remain flat.

Specimens for residual stress measurements were parted from the fatigue test bars by means of an abrasive cut-off wheel mounted on a horizontal-spindle surface grinder. Auxiliary experiments showed that the parting procedure employed did not measurably affect the grinding stresses in the test surfaces. Specimens were obtained from two locations in the test bar shown in Fig. 1. One type of specimen, called A, came from the larger grip end and had a section size of 2 by 2 in. The other type was sectioned so that its center was in the maximum stress region of the test bar. This was a B specimen and had a section size of 1.25 by 1.25 in. When

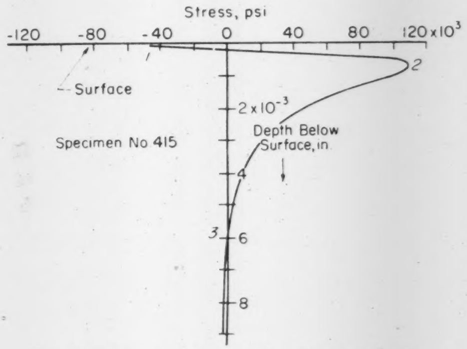

FIG. 3.—Typical Stress Distribution in Group AC Specimens.

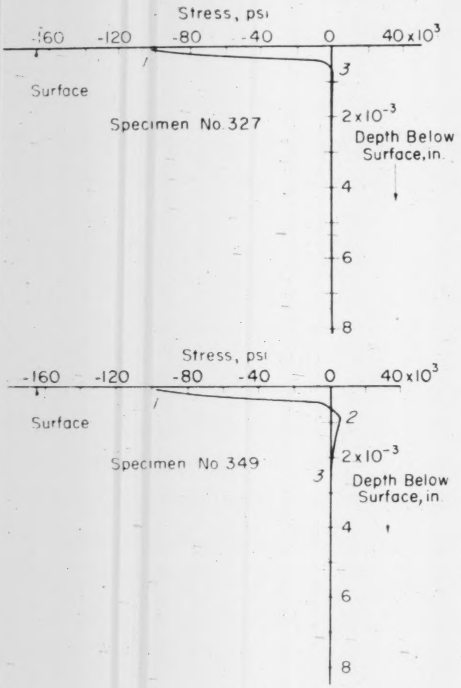

FIG. 4.—Typical Stress Distribution in Group AF Specimens.

this was sectioned from a broken test bar, the fracture surface served as one side of the square specimen.

It was hoped that any effect of cyclic stressing upon the residual stresses could be evaluated by comparing a B specimen, which underwent the maximum flexure, with an A specimen, which was not affected by cyclic stressing since it was clamped firmly in the grip of the fatigue testing machine and thus could not flex. A comparison of these two types of specimens taken from an untested bar in the AC group showed that the maximum residual tensile stresses were somewhat higher for the A than for the B specimens, but not enough to invalidate a study of the effect of cyclic stress upon the residual stresses. It is probable that the minor difference between the A and B specimens resulted from some variation in the effective grinding conditions with respect to the distance of the specimen edges from the ends and sides of the bars. Type B specimens from untested bars were used throughout to determine the effect of residual stresses upon the fatigue properties of the various groups of test bars.

TABLE II.—SURFACE ROUGHNESS AND RESIDUAL STRESS DISTRIBUTION DATA.

| Test Group | Type of Stress Distribution | Surface Stress, psi | Peak Tensile Stress, psi | Depth of Peak Tensile Stress, in. | Depth of Stress Disappearance, in. | Surface Roughness,[a] microinches AA |
|---|---|---|---|---|---|---|
| AA............ | 1 | $13 \times 10^3$ | $23 \times 10^3$ | $0.2 \times 10^{-3}$ | $0.8 \times 10^{-3}$ | 11 |
| AB............ | 2 | −25 | 18 | 0.2 | 0.7 | 11 |
| AC............ | 2 | −29 | 122 | 0.7 | 6.1 | 23 |
| AD............ | 2 | −67 | 12 | 1.0 | 3.8 | 11 |
| AE............ | 2 | −107 | 32 | 0.7 | 5.7 | 13 |
| AF............ | 3 | −96 | 1 | 0.8 | 0.8 | 11 |
| AG............ | 2 | −87 | 21 | 0.8 | 2.5 | 10 |
| BA............ | 2 | Comparable to AG Test Group | | | | |
| BC............ | 1 | 34 | 49 | 0.3 | 4.5 | 23 |

[a] Arithmetic average (calculated as 90 per cent of rms readings).

### *Typical Stress Patterns:*

Three general types of stress distributions were found for the grinding conditions investigated. The distinguishing features of these stress patterns are:

*Type 1.*—The residual stress is tensile at the surface. The peak tensile stress may occur slightly subsurface. Typical of this stress distribution are groups AA and BC (see Fig. 2).

*Type 2.*—The residual stress is compressive at the surface. A significant peak tensile stress occurs slightly subsurface. Typical of this stress distribution are groups AB, AC, AD, AE, AG, and BA (see Fig. 3).

*Type 3.*—The residual stress is compressive at the surface. Either a small peak tensile stress (less than 5000 psi) occurs slightly subsurface or no significant subsurface tensile peak is observed. Group AF is typical of this stress distribution (see Fig. 4).

In Figs. 2, 3, and 4, slanted numbers 1, 2, and 3 refer to the locations of the surface stress, the subsurface peak tensile stress, and the depth at which the stresses become of insignificant magnitude, respectively. What is termed the surface stress is actually the stress at an arbitrary depth of 0.00005 in. below the ground surface. This depth was selected because of the difficulties experience with some of the specimens in obtaining reliable stress values closer to the surface.

Values of these stresses and the corresponding depths are listed in Table II for each of the groups studied. These are average values for at least two surfaces. Also included in this table are surface roughness values, which were measured perpendicular to the grinding direction on a profilometer after the fatigue tests were completed.

The stress patterns described above resulted solely from the test grinding operations and did not contain any components ascribable to the previous operation in which the test bars had been ground to the intermediate thickness of 0.140 in. This was established by measuring the stress distribution in a bar processed to that stage. The stress at the surface was roughly 30,000 psi com-

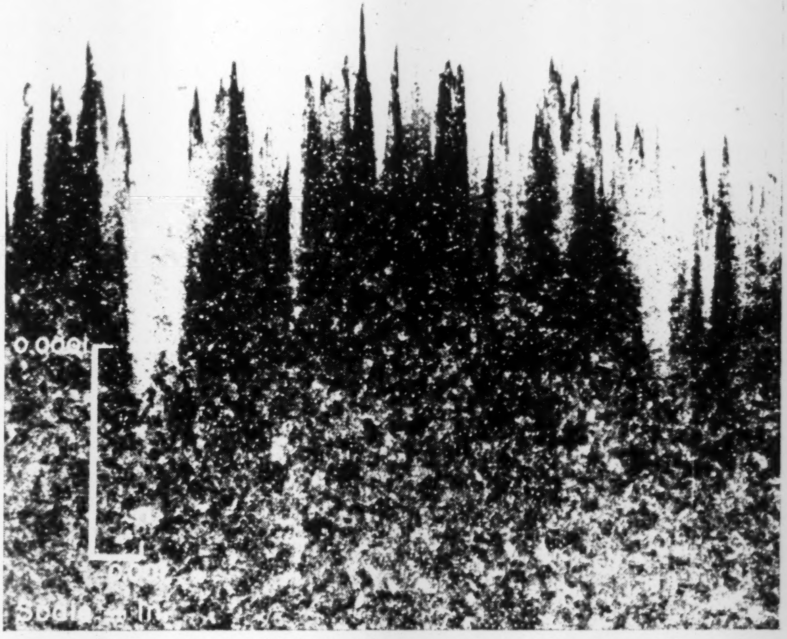

Fig. 5.—A Taper Section of Ground Surface of Typical Specimen of Group AC. Hyfercal Etch; Optical Magnification (×250); Taper Magnification (×44).

pressive, but it went through zero at 0.0005 in. below the surface to a maximum tensile stress of 3000 psi at 0.001 in. after which it dropped very gradually with greater depth to about 1500 psi tensile stress at 0.010 in. This stress distribution is shown in Fig. 8 of Reference (**2**). Since test grinding removed a layer of this thickness from each side, the previously existing residual tensile stress in what became the final surface of the test specimen amounted to only 1500 psi, which is negligible by comparison with the surface stresses present in the finished bars.

## GRINDING BURN

Heat generated in grinding can cause microstructural changes to take place in a surface layer whose thickness will vary with the severity of the operation. In addition, the surface may become discolored through the formation of an oxide film. Considerable microstructural changes can occur even in the absence of such discoloration or visible burn because under some grinding conditions the oxide film is ground away by the following portion of the wheel as soon as it is formed. None of the test groups were noticeably discolored but taper sections of representative specimens, such as are shown in Figs. 5, 6, and 7, revealed microstructural changes, which were extremely shallow for the most part.

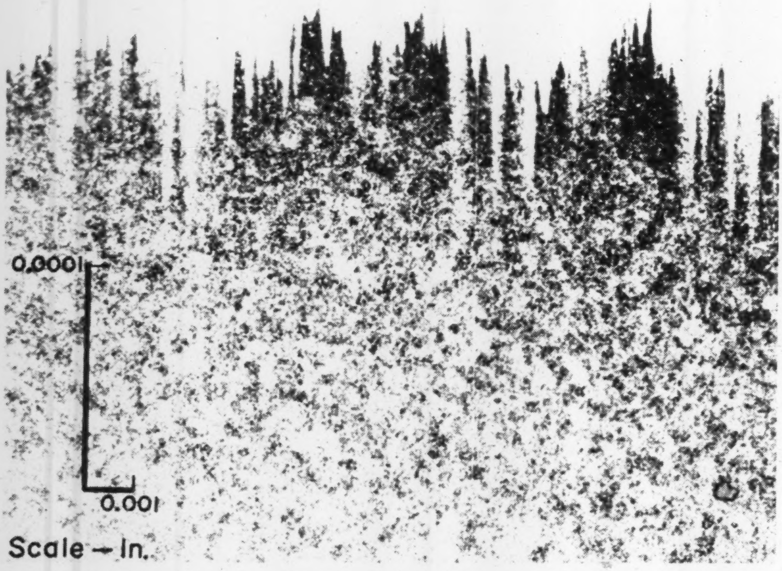

FIG. 6.—A Taper Section of Ground Surface of Typical Specimen of Group AF. Hyfereal Etch; Optical Magnification (×250); Taper Magnification (×47).

Several microstructural changes can occur in hardened steel when it has been burned in grinding. As shown by Tarasov and Lundberg (**4**), dark etching areas, brought out by normal etching procedures,[5] are caused by overtempering and white areas by rehardening. As brought out in Rowland's discussion of the foregoing paper, light-etching areas at the surface appear to be associated with deformation of the surface layer during chip formation.

These light-etching areas, which appear in all the taper sections (see Figs. 5, 6, and 7) should not be considered to be evidence of thermal rehardening. (The white area at the top of these taper sections is the nickel plate used to preserve the taper section in polishing.) The absence of such rehardening was established through both microhardness measurements and detailed metallographic examination.

The Knoop microhardness at 200 g load was essentially the same in some light-etching areas, which were of sufficiently large extent for indentations to be made, as in the very dark-etching areas immediately below, showing the former were softened by overtempering even though they did not darken when etched. Had there been rehardening, the microhardness in the light-etching areas would have been considerably higher than for the dark areas. Typical Knoop microhardness values for a specimen from group AB were 672 for the dark areas and 646 for the light-etching areas. These correspond to Rockwell hardness C 57 and C 56, respectively.

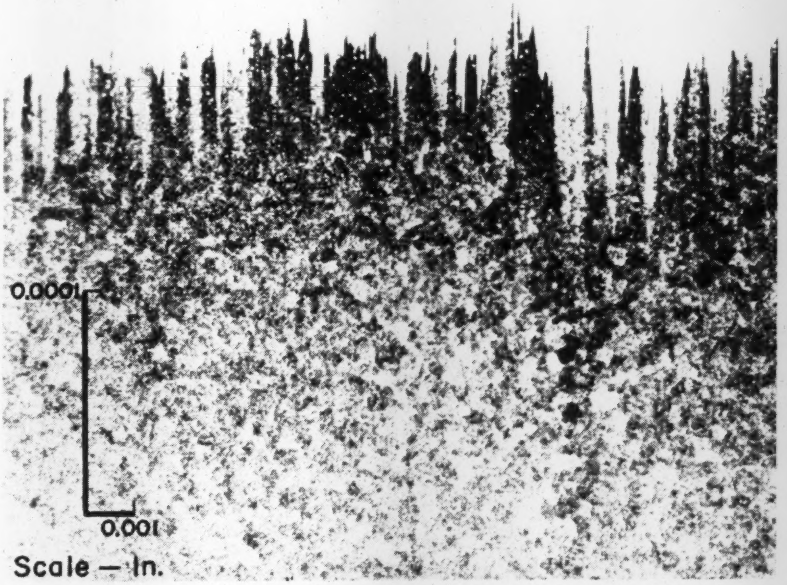

FIG. 7.—A Taper Section of Ground Surface of Typical Specimen of Group AG. Hyfercal Etch; Optical Magnification (×250); Taper Magnification (×47).

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[5] The hyfercal etchant, developed for the detection of the metallographic changes in hardened steel associated with grinding heat, consists of 2 ml concentrated HCl and 4 g $FeCl_3 \cdot 6H_2O$ in methyl alcohol to make 100 ml of reagent. Similar, but less clearcut results, would be obtained with a nital etch.

The taper sections also provide a visual indication of a microstructural nature that the light-etching areas cannot be attributed to thermal rehardening. Such rehardening is always associated with an underlying dark-etching zone of strongly overtempered martensite and the two are separated by a transition microstructure in which rehardening has occurred along the prior austenitic grain boundaries while the interior of the grain has been overtempered (4). There was no sign of this transition structure in any of the taper sections, and in some, like the one in Fig. 6, there was no darkening at all below the light-etching areas.

TABLE III.—SUMMARY OF FATIGUE RESULTS.

| Test Group[a] | Fatigue Limit (Maximum Applied Stress), psi | Standard Deviation, psi | Nucleation of Failure[b] |
|---|---|---|---|
| AA-*R*..... | $73 \times 10^3$ | $1.4 \times 10^3$ | Surface |
| AB-*R*..... | 70 | 9.8 | Surface |
| AC-*R*..... | 62 | 4.0 | Surface |
| AD-*R*..... | 73 | .... | Surface |
| AE-*R*..... | 70 | 1.2 | Surface |
| AF-*R*..... | 99 | 1.1 | Subsurface |
| AG-*R*..... | 91 | 3.5 | Subsurface |
| BA-*R*..... | 92 | 2.9 | Subsurface |
| BC-*R*..... | 73 | 1.3 | Surface |
| AA-*U*..... | $110 \times 10^3$ | .... | Surface |
| AC-*U*..... | 91 | 1.4 | Surface |
| AG-*U*..... | 133 | 3.5 | Subsurface |

[a] *U* denotes undirectional bending.
*R* denotes reversed bending.

[b] General trend of origin of failures for a group.

The taper sections show that the depth of the microstructural changes is only a small fraction of the depth to which grinding stresses penetrate. For example, in both Figs. 6 and 7 the depth of the microstructural changes is of the order of 0.0001 in., but the depths at which the residual stresses become negligible are 0.0008 and 0.0025 in., respectively. This indicates, therefore, that grinding heat is responsible for only a portion of the residual stress pattern and that the rest of it may be attributed to plastic deformation.

## FATIGUE STUDY

### *Equipment and Procedure:*

Fatigue specimens for nine grinding conditions were tested in reversed bending in a machine that deflected the specimen as a cantilever at a frequency of 1600 cpm (1). In addition, three of these groups of specimens were tested under unidirectional bending (zero to maximum deflection). Prior to testing, the sharp edges of each bar were gently polished with a fine abrasive cloth. Grip failures occurred in some of the specimens first tested, but they were completely eliminated when the grip ends were sufficiently strengthened by grit blasting. It was observed that some test bars were prematurely failing at tiny corrosion pits during humid weather conditions. To remedy this situation, the test bars were coated with a rust preventive prior to testing. The fatigue machine was also fitted with a cover, and silica gel was placed in an open container under the cover. These measures essentially eliminated this difficulty.

The load on all specimens was adjusted by means of a special dial gage device that measured directly the curvature of the specimen when the machine and specimen were run manually through a complete stress cycle. Small dynamic effects were taken care of in the calibration of the device. It was estimated that computed applied stresses were within 3 per cent of the true value.

In view of the limited number of samples available within each group, no more than five specimens were tested in the finite lifetime region to establish the approximate fatigue limit; the remainder of the specimens were tested in the fatigue limit region. Specimen runout was arbitrarily selected at 8,000,000 cycles.

In scheduling the tests in the fatigue limit region, the staircase method (5) was employed since this afforded a convenient method for concentrating the tests in the desired strength range. The stress interval used in this method was 3000 psi.

*Test Results:*

The results of the fatigue studies for each group were analyzed for a mean fatigue limit in accordance with the method of Dixon and Mood (6). The standard deviation also was computed for each group as an indication of the scatter in the fatigue limit range. The mean fatigue limits and standard deviations for each test series are listed in Table III, as well as the most frequently observed sites for fatigue nucleation for each test series (either at the surface or below the surface). *S-N* curves are not included because the data in the finite lifetime range were insufficient in most cases.

The table shows that the fatigue limits obtained under completely reversed stress vary between 62,000 psi and 99,000 psi. In the case of unidirectional stress,

TABLE IV.—EFFECT OF CYCLIC STRESS ON RESIDUAL STRESSES IN AC BARS.

| Bar | Lifetime, cycles | Specimen Surface | Peak Residual Tensile Stress, psi | |
|---|---|---|---|---|
| | | | A Specimen | B Specimen |
| No. 415 | Untested | Top | $124 \times 10^3$ | $110 \times 10^3$ |
| | | Bottom | 102 | 88 |
| No. 379[a] | 696 000 | Top | 164 | 157 |
| | | Bottom | 175 | 162 |

[a] Tested in unidirectional bending.

TABLE V.—EFFECT OF CYCLIC STRESS ON RESIDUAL STRESSES IN AC AND AF BARS.

| Group | Bar | Applied Stress, psi | Lifetime, cycles | Average Peak Residual Tensile Stress, psi |
|---|---|---|---|---|
| AC | No. 415 | Untested | ... | $100 \times 10^3$ |
| AC-*U* | No. 379 | $91 \times 10^3$ | 696 000 | 160 |
| AC-*U* | No. 300 | 91 | Runout | 130 |
| AC-*R* | No. 309 | 64 | 600 000 | 120 |
| AC-*R* | No. 543 | 64 | Runout | 100 |
| | | | | Average Max Residual Compressive Stress, psi |
| AF | No. 327 | Untested | ... | $90 \times 10^3$ |
| AF-*R* | No. 518 | $99 \times 10^3$ | 79 000 | 95 |
| AF-*R* | No. 609 | 99 | 1 350 000 | 100 |
| AF-*R* | No. 668 | 99 | 8 406 000 | 120 |
| AF-*R* | No. 349 | 99 | Runout | 90 |

the range in fatigue limits was between 91,000 psi and 133,000 psi. The significance of these large ranges in fatigue limit is discussed later in conjunction with the residual stress studies.

It might be considered that the fatigue limits obtained are low, since the material was heat treated to Rockwell hardness C 59, corresponding to a tensile strength probably greater than 300,000 psi. However, the previous investigation (1) showed that the fatigue limit of gently ground flat bars of the same steel, shape, and hardness was 65,000 psi. On the other hand, the fatigue limit for similarly processed round rotating-beam specimens was 140,000 psi. Thus, the apparently low fatigue limits obtained in the present study are in line with those previously obtained and are probably caused by the differences in section shape and size, and differences associated with the two types of fatigue tests.

It was necessary to determine whether the second hardening treatment to which excessively warped specimens had been subjected might influence the fatigue properties of the steel. Two groups of specimens, AG (hardened once) and BA (hardened twice) were ground under identical conditions. The fatigue limits for reversed bending were practically the same, being 91,000 and 92,000 psi, respectively. Thus, it was not necessary to establish a new base-line for the doubly heat-treated bars.

## DISCUSSION

### *Effect of Cyclic Stressing on Residual Stresses:*

Evidence concerning the effect of cyclic stressing on the magnitude of the residual stresses comes from a series of studies on tested and untested bars from groups AC and AF. Pertinent data upon which subsequent discussion is based are listed in Tables IV and V.

Consider first Table IV, in which are presented the peak residual tensile stresses for each surface of two bars (one untested, the other tested), as obtained on both A and B specimens. In bar 415, which was not tested, there is a difference of about 14,000 psi in peak tensile stress between specimens sectioned from grip ends (A specimens) and specimens from the center (B specimens), the former having the higher stress. Examination of the data for bar 379, tested under unidirectional bending (top surface cyclically stressed in tension, bottom surface only in compression), discloses that the difference between peak residual tensile stresses for unflexed A specimens and for the flexed B specimens was quite small. This difference was of the same general magnitude as the difference noted for specimens from the same two locations in the untested bar. Moreover, the residual stress curves retained their original shape.

The data listed in Table V show that the peak residual tensile stresses in all the tested AC bars were equal to or greater than those in the untested bar. The second part of Table V shows that the maximum compressive residual stresses in the tested AF bars were equal to or greater than those in the untested bar. Again, the residual stress curves had the same shape in both tested and untested bars. Since cycling at the fatigue limit would not be expected to increase the magnitude of the residual stresses, their higher values in some of the tested bars is evidence of the scatter that may be obtained by grinding.

On the strength of these observations, it appears that cyclic stressing of the hardened bars did not cause any reduction in the magnitude of the initial residual stresses.

### *Scatter in Fatigue Results:*

Even with the carefully controlled test grinding employed in this study,

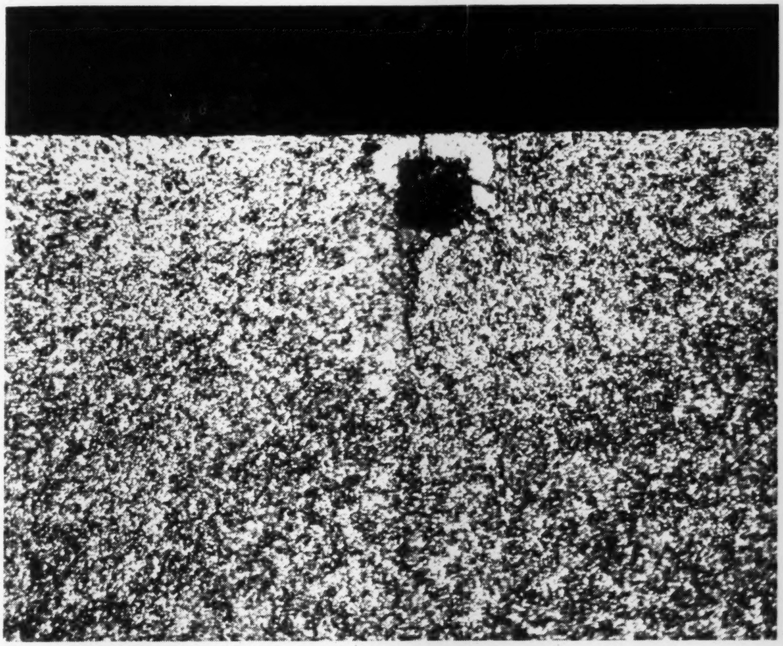

FIG. 8.—Typical Subsurface Nucleation at Oxide Inclusion (×250).

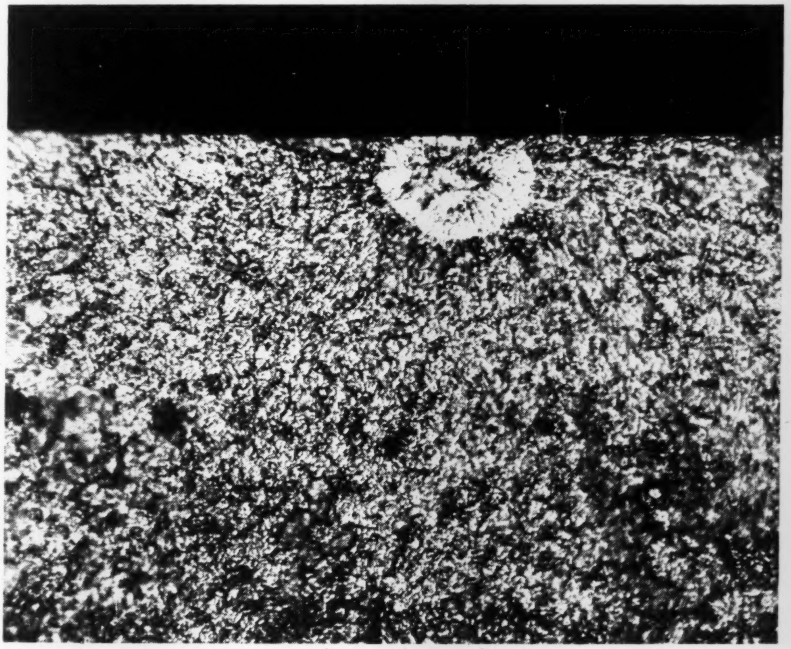

FIG. 9.—Typical Subsurface Nucleation at a Titanium Cyanonitride Inclusion (×500).

appreciable scatter in residual stresses was encountered. This scatter appears to be responsible for some of the scatter in the fatigue results. In Table V, of the two AC-*U* bars tested at an applied maximum stress of 91,000 psi, the bar with the lower residual tensile stress did not fail, while the other lasted only 696,000 cycles. A similar relationship was observed for the two AC-*R* bars.

Another factor is present in the case of the AF-*R* bars tested at a maximum stress of 99,000 psi (see Table V). For these, the tabulation shows some variation in the surface compressive stress. All of the bars, however, had about the same subsurface tensile stress (less than 5000 psi). Failure in this group of bars was generally subsurface in origin and usually at an oxide or titanium cyanonitride inclusion (see Figs. 8 and 9). It is interesting to note that bar 518 in the foregoing tabulation was an exception for this group of bars, in that failure occurred at the surface at an inclusion. This bar had the shortest life of the four bars tested at the stress level of 99,000 psi.

TABLE VI.—SUMMARY OF GRINDING CONDITIONS AND FATIGUE RESULTS FOR REVERSED BENDING.

| Group | Grinding Conditions | | | Fatigue Limit at $8 \times 10^6$ cycles, psi |
|---|---|---|---|---|
| | Grinding Fluid[a] | Wheel Hardness | Unit Downfeed, in. | |
| AC | Sol. B | M | $2.0 \times 10^{-3}$ | $62 \times 10^3$ |
| AA | Sol. B | H | 0.1 | 73 |
| AB | Sol. B | H | 0.1 | 70 |
| BC | Sol. F | I | 1.0 | 73 |
| AD | Gr. I | M | 1.0 | 73 |
| AE | Gr. I | M | 2.0 | 70 |
| BA | Gr. L | I | 1.0 | 92 |
| AG | Gr. L | I | 1.0 | 91 |
| AF | Gr. I | I | 1.0 | 99 |

[a] Sol = soluble oil; Gr = grinding oil.

Attention in the literature has been given recently to the predominant influence of inclusions in engineering materials on scatter in fatigue life. The observations in this investigation bear out that metallurgical discontinuities are important in this regard. However, it appears that some of the scatter in fatigue results also may come from normal bar-to-bar variations in residual stress that arise during careful preparation.

### *Influence of Grinding Conditions on Fatigue Strength:*

In the time available for this investigation, only a few grinding variables could be studied. Therefore, the effect of grinding conditions on fatigue strength can be described only in general terms. The grinding conditions studied included those used in commercial practice as well as extreme conditions. The results of the limited study of these are considered to be of practical use in indicating how the fatigue properties of hardened steel may be affected by certain surface grinding conditions. Table VI combines the pertinent data from Tables I and III of grinding conditions and fatigue results for the reversed bending tests.

In the previous investigation by Tarasov and Grover (1), comparison of the two groups of bars of Rockwell hardness C 45 (one, gently ground; the other, gently ground and electropolished), showed the two groups to have the same fatigue limit. In the same investigation, bars having Rockwell hardness C 59 were prepared using the same gentle grinding procedure as for the softer bars. These had a fatigue limit of 65,000 psi. The data on the softer bars suggest that electropolished bars of Rockwell hardness C 59 also might have had a fatigue limit of about 65,000 psi.

In the present study, groups AA and AB were gently ground similarly to those of the preceding study. The observed fatigue limits of 73,000 psi and 70,000 psi are not too dissimilar from the 65,000 psi previously obtained (considering normal heat-to-heat variations in fatigue strength). Hence, the base fatigue limit for this steel, specimen configuration, and type of test is considered to be 71,500 psi.

In Table VI, it is seen that specimens ground under commercial grinding conditions with soluble oil (group BC) had essentially the same fatigue strength as the two base-line groups (AA and AB), ground with a softer wheel and a unit downfeed of 0.0001 in.

Again considering soluble oil grinding, quite severe grinding conditions (group AC bars harder wheel, M grade; heavier downfeed, 0.002 in.) resulted in a decrease in fatigue limit from the base value of about 13 per cent.

In using straight grinding oils with I grade wheels, increases in fatigue strength over the base-line value up to about 38 per cent were noted (groups AG, BA, and AF). However, the two groups ground with the harder (M grade) wheel (AD and AE) had fatigue strengths about the same as AA and AB, indicating the unpredictable nature of the effects of the various grinding variables when taken in combination.

The comparison of groups BA and AG with group AF shows that different kinds of straight grinding oils can produce significant differences in fatigue strength.

The only data for assessing the effect of unit downfeed are those for groups AD and AE. Although the results for AD and AE indicated the same fatigue limits, this is not expected to occur under normal grinding conditions. Both sets of bars were ground with an M grade wheel with grinding oil. Under commercial grinding conditions, a sufficient increase in unit downfeed might well cause some decrease in the fatigue limit.

### *Influence of Residual Stresses on Fatigue Strength:*

At the present time, there is considerable interest in the effect of machining and heat treating on the production of residual stresses in machine components and their subsequent effect upon fatigue behavior. On the basis of past work, it is generally believed that residual stresses (as well as other factors) affect fatigue properties—surface tensile residual stresses decreasing fatigue strength, surface compressive residual stresses increasing fatigue strength. In some of this previous work—but by no means all of it—the residual stresses have been inferred or determined experimentally on the basis of sectioning techniques (and others) not capable of achieving the fineness of detail that Letner's (2,3) studies have indicated necessary in the case of grinding stresses in hardened steel. Thus, some of the generalizations that have been made concerning residual stresses and fatigue data bear further critical review. The data obtained in this investigation permit some analysis of these factors; however, it is expected that further studies will be necessary to resolve a number of questions raised by this investigation.

The results of this study have been

examined in a number of ways. One of these is illustrated in Fig. 10 which is a plot of reversed bending fatigue limit against peak tensile residual stress. A plot was made also of fatigue limit *versus* residual stress at the surface; however, the graph in Fig. 10 showed less scatter.

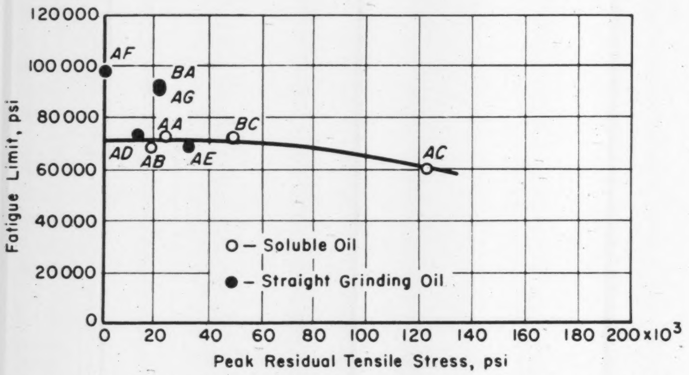

FIG. 10.—Influence of Peak Residual Tensile Stress on Reversed Bending Fatigue Limit.

From this figure, it appears that all of the open circles and two of the solid circles can be represented by the curve drawn on the graph; whereas three of the solid circles fall well above the curve.

In considering first the data fitting the curve, in each case failures initiated at or near the surface. In all but groups AA and BC, the surface stresses were compressive; however, there was a marked tensile peak evident within the first 0.001 in. In the case of AA and BC, the surface stress was tensile with a subsurface tensile stress peak at a depth of about 0.0002 in.

It appears that surface stresses alone, whether compressive or tensile, are not sufficient to determine whether improvement or reduction in fatigue strength will occur. Other features in the residual stress picture are important as well as other factors discussed later.

It is evident for the grinding conditions and steel studied in this investigation that there is a range of residual tensile stress values for which the fatigue limit remains at the base-line value of about 71,500 psi. A moderate decrease is observed for high values of residual tensile stress as illustrated by the point for group AC.

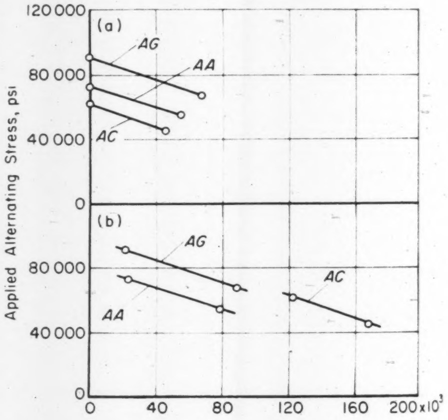

FIG. 11.—Stress Range Diagram for 8 × 10⁶ Cycle Lifetime.

(*a*) Mean Applied Stress, psi.

(*b*) Mean Applied Stress Plus Peak Tensile Residual Stress, psi.

The data represented by the upper three solid circles are for bars in which failure occurred primarily subsurface. For these, the surface stresses were compressive, but moderate residual tensile stresses were observed in two of the three groups at a distance of about 0.001 in. below the surface. These bars, ground with straight grinding oil and a moderately soft wheel, were apparently strengthened by this type of grinding relative to the bars whose data points cluster around the curve. This is illustrated by comparing the results for bars of groups AE and AG (ground with straight grinding oil).

| Test Group | Surface Residual Stress, psi | Peak Tensile Residual Stress, psi | Fatigue Limit, psi |
|---|---|---|---|
| AE..... | $-107 \times 10^3$ | $32 \times 10^3$ | $70 \times 10^3$ |
| AG.... | $-87$ | 21 | 91 |

The fatigue limit was 21,000 psi higher for the AG bars than for the AE bars, which failed below and at the surface, respectively, but the residual stresses were effectively the same, as can also be deduced from the corresponding data for the AD bars (Tables II and III). Thus it appears that these differences in fatigue behavior were caused by some difference between the ground surfaces with respect to the nature of the mechanical deformation caused by grinding.

There are other ways of examining the data obtained in this investigation. Since three groups of bars (AA, AC, and AG) were tested under conditions of reversed bending and unidirectional bending, the data obtained from these tests were plotted on a stress range diagram (modified Goodman diagram) with coordinates of alternating and mean applied stress. This is shown in Fig. 11(*a*). The constant lifetime lines drawn between corresponding points are for $8 \times 10^6$ cycles, and they turned out to be nearly parallel over the limited range of load ratio investigated.

Now if the residual stress is considered to be equivalent to a mean applied stress, then changing the abscissa to correspond to the sum of these two stresses should shift the three lines in such a way that all the points lie on a single curve, provided that the correct value of the residual stress is selected and that other factors do not affect the results. Such a plot (Fig. 11(*b*)), in which the peak residual tensile stress has been added to the mean applied stress, indicates that these assumptions are not justified. A similar plot, using the surface residual stress in place of the peak tensile one, changed the relative positions of the three lines but did not bring them together any better.

Thus the residual stresses obtained in this study cannot be added to the mean applied stresses for the purpose of predicting useful fatigue limits for various combinations of maximum and minimum applied stresses. Only for the reversed bending tests were enough data obtained to establish the relationship shown in Fig. 10 between the fatigue limit and the residual grinding stress. Not nearly enough data were available for doing the same for the unidirectional tests, and all that can be safely said is that the fatigue limit for unidirectional bending was affected by the grinding conditions and the resultant residual stresses in about the same way as that for reversed bending. To find out how to combine properly residual stresses with mean applied stresses in a Goodman-type diagram, if this is not rendered impractical through the interference of other factors like cold work, will require considerably more experimentation.

## CONCLUSIONS

The conclusions of this study are based upon the reversed bending fatigue limits obtained with flat test bars made from modified AISI 52100 steel uniformly hardened to Rockwell C 59 and ground longitudinally to produce a variety of residual stress distributions resulting only from the grinding operations. The results should be equally applicable to other uniformly hardened steels in the same hardness range, but not necessarily to case-hardened steels nor to steels containing high heat-treating stresses.

The fatigue failures generally nucleated at the surface in the usual manner, but an exception was noted when a moderately soft wheel (I grade) was used with either of two grinding oils. Here the failures nucleated at subsurface inclusions and the fatigue limits were appreciably higher than for surface nucleation.

When nucleation occurred at the surface, the fatigue limit fell on a smooth curve if plotted against the highest residual tensile stress (which was located just below the surface), but not if plotted against the surface stress, which ranged from compressive to tensile, depending on the grinding conditions. The fatigue limit was not affected measurably by the peak residual tensile stress until this exceeded 50,000 psi, and even when this residual stress was in the vicinity of 120,000 psi, the fatigue limit was only 13 per cent lower than the base-line value.

Subsurface nucleation of failures and the associated higher fatigue limits could not be attributed to compressive surface stresses alone because under somewhat different grinding conditions that happened to produce very similar stress distributions, the failures nucleated at the surface and the fatigue limits fell on the base line. It appears that under certain grinding conditions the surface can be strengthened sufficiently by mechanical deformation of a beneficial sort to cause fatigue failure to initiate somewhat below the surface at some inclusion.

Not nearly enough grinding conditions were investigated to permit definite predictions to be made about the effects of the numerous grinding variables upon the fatigue limit. Evidence was obtained, however, that the fatigue limit is not reduced by good commercial grinding practice below the base-line value obtained under very gentle grinding conditions, and that even under severe grinding conditions, the reduction is only moderate.

Grinding conditions had a similar effect upon the fatigue limits for unidirectional bending as for reversed bending, but it was not found possible to combine the residual stresses with the mean applied stresses on a Goodman-type diagram.

Cyclic stressing did not cause any measurable reduction in the magnitude of the residual grinding stresses.

Some of the scatter in the fatigue results appeared to be associated with the variation in the residual stresses within a group of test bars which occurred in spite of the precautions taken to reproduce the grinding conditions as closely as possible. Other scatter appeared to be associated with the presence of non-metallic inclusions.

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## REFERENCES

(1) L. P. Tarasov and H. J. Grover, "Effects of Grinding and Other Finishing Processes on the Fatigue Strength of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 668–698 (1950).

(2) H. R. Letner, "Residual Grinding Stresses in Hardened Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 77, pp. 1089–1098 (1955).

(3) H. R. Letner, "Influence of Grinding Fluids Upon Residual Stresses in Hardened Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 79, pp. 149–153 (1957).

(4) L. P. Tarasov and C. O. Lundberg, "Nature and Detection of Grinding Burn in Steel," *Transactions*, Am. Soc. Metals, Vol. 41, pp. 893–927 (1949).

(5) J. T. Ransom and R. F. Mehl, "The Statis-

tical Nature of the Fatigue Properties of SAE 4340 Steel Forgings," Am. Soc. Testing Mats. (1953). (Issued as separate publication *ASTM STP No. 137.*)

(6) W. J. Dixon and A. M. Mood, "A Method of Obtaining and Analyzing Sensitivity Data," *Journal Am. Statistical Assn.*, Vol. 43, March, 1948, pp. 109–126.

## DISCUSSION

MR. J. D. MARBLE.[1]—Mention is made of the possibility of getting a surface that had no residual stress. Was it planned to produce any specimens with electropolishing or something similar for comparison so that this study might then be essentially complete? Perhaps someone would be willing to support the remainder of such a program. One other question related to specimens which failed. Might one use the mean stress at the position where failure took place rather than the surface.

MESSRS. G. M. SINCLAIR AND JODEAN MORROW[2] (*by letter*).—The authors have provided a most interesting and informative set of data on the effect of grinding conditions on fatigue strength of high hardness steel. We have been conducting a study on the significance of residual stress on fatigue strength of SAE 4340 steels at various hardness levels by considering residual stress analogous to mean stress. Consideration of the similarities and differences in conclusions may be of further interest. First the writers would agree with the conclusion that cyclic stressing does not appreciably reduce the magnitude of residual stress in steels having a Rockwell hardness C-55 or greater. Our measurements of change in mean stress under conditions of constant amplitude of cyclic strain and constant mean strain indicate that the mean stress decays or "washes out" most readily in the softer steels.

Figure 10 suggests that the peak tensile residual stress is not very effective in lowering the fatigue limit of high hardness steel. It is believed, however, that this conclusion may be somewhat misleading due to the fact that the residual stresses discussed in the paper had an extremely sharp gradient—in some cases, changing from compression to a maximum value in tension in less than 0.001 in. from the surface. When the sharp gradient in residual stress is taken into consideration, the problem assumes some of the aspects of reduction in fatigue strength caused by very small sharp notches. It has been demonstrated in the past that the reduction in fatigue strength caused by such notches is far less than would be predicted from the theoretical stress concentration factor based on elasticity concepts. It has been suggested that this is due to the fact that the depth of stress gradient at the root of very small notches is of the same order as the strength determining elements of the microstructure (grain size, etc.).

Loss of fatigue strength due to large tensile residual stresses distributed over a sizeable depth from the surface has been adequately demonstrated in the past and further discussion by the authors relating to the possible influence of stress gradient caused by the grinding operations would be appreciated.

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[2] Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

Values of fatigue limit for this material having either electrolytically polished or mechanically polished test surfaces would also be of interest, if available.

MR. R. L. MATTSON[3] (*by letter*).—One question that comes to my mind is one related to the possible variation in residual stresses obtained with any one grinding procedure. Has enough work been done in this field to establish the point that a given grinding procedure and a given material will produce like stresses within say 25 per cent? It seems important to me in the appraisal of the possible influences of residual stresses that we control their magnitude just as we do any other variation in a test. The load stresses during fatigue cycling were held within 3000 psi. I am sure the variation in residual stresses was much greater. The paper gives the average residual stress obtained from at least two surfaces and Table IV shows a range of variation in like specimens from 88,000 psi peak tensile stress to 175,000 psi. Information on this might be helpful in the appraisal of the experiment.

Noting that the corners were stoned causes me to wonder if the fatigue testing may have tended to evaluate the effect of stoning rather than grinding. Information on the location of the origin of the failures with respect to these critical corners might clear up this question.

Standard deviations are given for endurance limits which are considerably less in some instances than the stress interval used in the staircase testing method. An explanation of this would be appreciated since this spread is much narrower than our work has indicated.

It is noted that some of the residual stress analyses were made of both the gripped portion of the specimen and the portion subjected to fatigue cycling and that some consistent difference was found. Could this possibly, in part, be attributed to the cold working effect of the grips?

Since both transverse and longitudinal residual stress data were taken, it would appear feasible to check out the validity of the distortion energy criterion of fatigue. Seldom do we have such a good picture of both residual stress state and applied load conditions.

I am sure that the authors feel that much additional work is needed before there is a satisfactory clarification of importance and possible significance of the complex and apparently highly variable triaxial residual stress state and the still unexplained fatigue phenomenon.

MR. J. G. ROBERTS[4] (*by letter*).—I would like to comment on the origin of the residual stress induced by grinding. The grinding process may be roughly described as a severe sliding process. Bowden and Thomas have measured very high temperatures ("hot spots") that occur in sliding.[5] These hot spots occur under the following conditions: steel sliding on glass at 700 cm per sec, 350 g normal load, maximum temperature 900 to 1000 C, irradiating area $0.25 \times 10^{-3}$ and $0.15 \times 10^{-3}$ sq cm respectively, with a build-up time $3 \times 10^{-3}$ sec. Their analysis indicates that the temperature at the contact area is mainly a function of the normal load and sliding velocity. Bowden and Ridler[6] theoretically developed and experimentally verified that the effect of lubrication is to lower the temperature of "hot spots" to $\frac{1}{3}$ to $\frac{2}{3}$ of that encountered with no lubrication. From this, it seems that residual stress due to grinding have their origin in high temperatures developed and associated structural changes in rehardening and retempering.

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[4] Engineering Mechanics Department, Research Staff, General Motors Corp., Detroit, Mich.

[5] F. P. Bowden and P. H. Thomas, "The Surface Temperature of Sliding Solids," *Proceedings*, Royal Soc. of London, Vol. 223, April 1954, p. 29.

[6] F. P. Bowden and K. E. W. Ridler, "Physical Properties of Surfaces," *Proceedings*, Royal Soc. of London, Vol. 154, May, 1936, pp. 640–656.

It would appear that the endurance limit for group AG tested in reversed bending might be low (91,000 psi) because of unusual heat treatment caused by grinding. It would be interesting to see the endurace limit of carefully ground and electropolished (approximately 0.002 in. metal removed) specimen.

MESSRS. W. S. HYLER, L. P. TARASOV, AND H. R. LETNER (*authors' closure*).—Replying to Mr. Marble, in planning our test program, we considered the advisability of preparing a group of electropolished test specimens in order to establish the fatigue limit for essentially the stress-free condition. We decided against this since we had previously found[7] that the fatigue limit was approximately the same for the very gently ground, the mechanically polished, and the electropolished conditions, and we thought that it would be more profitable to investigate other variables in the time we had available. This reasoning appears to be justified by the horizontal nature of the curve in Fig. 10. We have completed the preparation of all the specimens that we plan to test, but a number of unfinished test bars are available for processing if anyone wants to investigate the stress-free condition.

When failure nucleates below the surface, as it did in many instances because of the grinding conditions, the mean stress is not the same at that point as at the surface both because the residual stress is different and because the inclusion associated with nucleation is a stress raiser. Thus the mean stress defined in this manner does not have a numerical value that can be readily determined. If instead of the mean stress, we consider the actual applied stress at the failure nucleus, this is slightly lower than the corresponding stress at the surface of the test specimen. The points on an *S-N* diagram would plot a little lower by amounts depending on the depth of nucleation. However, it would not be possible to evaluate the fatigue limit by the staircase method since this is based on the constancy of the applied stress interval between successive test specimens, and this interval would fluctuate as the depth of nucleation varied from one specimen to the next.

In reply to Messrs. Sinclair and Morrow, it is quite possible that the fatigue properties might be affected by a deeply penetrating residual stress but not by a shallow one of the same magnitude. Our conclusions about the relationship between residual stresses and fatigue properties are limited to *grinding* stresses in *uniformly hardened* steel, and such stresses have repeatedly been found to be very shallow.

Further experimentation is needed to determine the extent, if any, to which the fatigue properties may be affected by the steepness of the stress gradient perpendicular to the surface. In such a study, it would be important to avoid cold working the surface since this may partly or completely overcome the effect of residual stress on the fatigue properties when the stress is shallow but not when it is deep.

Mr. Mattson's question about the reproducibility of grinding stresses can be answered only in a general way. Under reasonably good grinding conditions, the reproducibility of the residual stress curves was generally very good as judged from comparisons of the top and bottom surfaces of a given specimen, which were ground independently. But when extreme grinding conditions were used, either severe (AC) or gentle (AA), the reproducibility was poor. In these instances, it was necessary to investigate additional specimens in order to get a better idea of the residual stresses that might be present for those grinding conditions, and this in itself increased the probability of obtaining a wider spread of results than when only two surfaces were studied. The difficulty of getting reproducible grinding stresses under extremes of grinding conditions is paralleled by similar experience with other grinding variables such as wheel wear and power consumption. In fact, this is not limited to grinding but is undoubtedly true of other metal-processing operations. From a practical standpoint, it did not really matter that the residual stresses for certain grinding conditions were poorly reproducible, inasmuch as they had relatively little effect upon the fatigue limit, as shown both by the curve in Fig. 10 and by the reasonably narrow width of the fatigue limit bands.

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[7] See reference (**1**) of paper.

The standard deviations for the fatigue limits given in Table III were computed by the procedure developed by Dixon and Mood for the staircase method. Some of the values are lower than would be expected from the stress interval used. This might be attributed to the small population employed in the analysis. However, it is believed the method of computing itself is not exactly rigorous. Perhaps it would have been better to omit the standard deviation values from Table III. However, it was believed that their inclusion would provide a relative measure of the scatter in the data and not an absolute measure of the standard deviation.

The edges of the test specimens were not stoned but were very lightly abraded with fine abrasive paper to remove the wire edges. A detailed study of the distribution of failure origins with respect to the edge is being prepared for publication. This study has shown conclusively that fatigue failures can originate at any distance from the edge and that the barely perceptible rounding of the edges does not influence the results.

The differences between the residual stresses in the grip and center portions of the test specimens were essentially the same for tested and untested bars. Thus the grips did not affect the residual stresses already present in the bars, but they may have had some slight cold-working effect.

Although the sliding process discussed by Mr. Roberts must take place in any chip-forming process, the nature and magnitude of the effects involved can best be understood from an examination of the results actually obtained in grinding and not from comparison with the results of sliding experiments. Very careful metallographic taper-section studies showed that the grinding heat was sufficient in some instances to soften a very thin surface layer, but no thermal rehardening was observed. The depth of a typical softened layer, as determined metallographically, was much less than the depth of the residual tensile stress peak. Thus the tensile stress cannot be attributed to the microstructural changes involved in such tempering. The stress could well have been due to temperature changes which lasted too short a time to affect the microstructure. It should also be remembered that similar tensile grinding stresses can be produced in pure metals, where no microstructural changes can occur. Compressive grinding stresses cannot be readily explained in terms of temperature effects and are most likely associated with plastic deformation.

With regard to the AG test specimens, Fig. 7 shows clearly that the softened layer was only about 0.0001 in. deep, and this group of specimens can hardly be characterized as having been subjected to unusual heat treatment in grinding. The fatigue limit was much higher than for the specimens falling on the curve of Fig. 10, and was exceeded only by the AF specimens, which were the only ones in which the grinding stresses were entirely compressive.

# FRETTING FATIGUE STRENGTH OF TITANIUM ALLOY RC 130B*

By H. W. Liu,[1] H. T. Corten[1] and G. M. Sinclair[1]

## Synopsis

An investigation was made to determine the influence of a number of variables on the fretting fatigue strength of RC-130B titanium alloy. The variables studied included different gripping materials, hardness of the gripping materials, gripping pressure, surface preparation of specimens, dry lubricants, metallic coatings and special screen gripping shims.

An analysis of experimental results suggests that the primary mechanism responsible for fretting fatigue damage is the repeated frictional shear stress on the asperities or surface "high spots" which are in contact.

Based on prevention of fatigue crack initiation by this mechanism, a mathematical expression is proposed which relates the fretting fatigue strength to the fatigue limit of the specimen material, the hardness of the gripping material, and the coefficient of friction.

Fretting or fretting corrosion are terms commonly employed to describe the surface damage that occurs when two members, pressed into contact, undergo small repeated sliding or relative motion. Shrink or press fits, bolted or riveted joints subjected to repeated loads and ball bearings subjected to small oscillatory motion, are just a few examples of members that often exhibit surface damage and debris dust due to fretting. The fretted surface exhibits characteristics very similar to wear, including abrasion, metal transfer, and often increased roughness and pitting (**1,2**).[2] In addition, chemical oxidation products (debris and compacted surface layers) generally accompany fretting in air (**2**).

Fretting damage to a metal surface results in a reduced fatigue strength. In the past, this reduction in strength has been taken into account by employing a liberal factor of safety. Recent emphasis on the strength to weight ratio, introduction of new materials, and more severe operating conditions in the aircraft and automotive industries, all accentuate the importance of the fretting fatigue problem.

General agreement has not been reached with regard to the relative contributions of mechanical and chemical action in causing fretting damage. Chemical (**2,3**), mechanical (**1,4**), and combined chemical and mechanical mechanisms (**5**) have been given as the primary cause of fretting. Fretting damage has been measured in terms of weight loss (**5**),

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Research Assistant, Research Associate Professor, and Research Associate Professor, respectively, University of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 640.

TABLE I.—CHEMICAL COMPOSITION OF MATERIALS, PER CENT.

| Material | Aluminum | Magnesium | Vanadium | Silicon Maximum | Sulfur | Phosphorus | Manganese | Copper | Lead | Zinc | Chromium | Carbon | Iron Maximum | Nickel | Titanium | Other |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Titanium (RC130B) | 4.0 | .. | .. | .. | .. | .. | 4.0 | .. | .. | .. | .. | 0 to 10 max | .. | .. | Balance | Nitrogen 0.02 |
| Magnesium | .. | 99.0+ min | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. | .. |
| Aluminum (1100-F) | Balance | .. | .. | Silicon + Iron 1.0 max | .. | .. | 0.05 max | 0.02 | .. | 0.10 max | .. | .. | Silicon + Iron 1.00 max | .. | .. | .. |
| Aluminum (2011-T8) | Balance | .. | .. | .. | .. | .. | .. | 5.5 | 0.5 | .. | .. | .. | .. | .. | .. | Bismuth 0.5 |
| Aluminum (7075-T6) | Balance | 2.1 to 2.9 | .. | 0.50 | .. | .. | 0.30 max | 1.2 to 2.0 | .. | 5.1 to 6.1 | 0.18 to 0.41 | .. | 0.70 | .. | 0.20 max | .. |
| Copper | .. | .. | .. | .. | .. | .. | .. | 99.9+ min | .. | .. | .. | .. | .. | .. | .. | .. |
| Brass (70–30) | .. | .. | .. | .. | .. | .. | .. | 69.71 | 0.07 | 30.21 | .. | .. | 0.01 | .. | .. | .. |
| Lapelloy | .. | .. | 0.29 | 0.48 | 0.011 | 0.016 | 1.15 | .. | .. | .. | 11.12 | 0.28 | Balance | 0.12 | .. | Molybdenum 2.84 |
| Steel SAE (4340) | .. | .. | .. | 0.2 to 0.35 | 0.04 max | 0.04 max | 0.6 to 0.8 | .. | .. | .. | 0.7 to 0.9 | 0.38 to 0.43 | Balance | 1.65 to 2.0 | .. | .. |

severity of surface damage by visual observation (**1,2**), and reduced fatigue strength (**6,7,8**).

In terms of reduced fatigue strength, the fretting damage increases rapidly as the gripping pressure is increased in the low-pressure range. At gripping pressures greater than 5000 psi, the fretting damage remains fairly constant (**8**). Changes of geometric shape of the specimen may result in a beneficial effect (**6,9**), and cold working of the specimen surface is also a partial remedy for alleviating fretting fatigue damage (**6,7,9**), but lubrication has not been effective (**6**).

TABLE II.—SURFACE PREPARATION OF SPECIMENS.

| Series Number | Surface Finish | Preparation Process |
|---|---|---|
| 1 to 16 | Mechanically polished | (1) shaped (2) polished with 240, 1, and 00 polishing cloths successively. |
| 17 | Oxidized | (1) mechanically polished as described above. (2) batch No. 1, submerged in 30 per cent $H_2O_2$ for 2 hr at room temperature (3) batch No. 2, submerged in $H_2O_2$ for 16 hr at room temperature. |
| 18 | Shot peened | (1) mechanically polished as described above (2) shot peened by P39 Steel shots at 75 psi air pressure for 3 min. |
| 19 | Shot peened | Same as series 18, but P19 steel shot was used. |
| 20 | Teflon coated | (1) mechanically polished as described above (2) teflon sprayed on specimen, then baked at temperature of 400 F |
| 21 | Tungsten carbide plated | (1) mechanically polished as described above (2) tungsten carbide particles were carried by a supersonic wave and were embedded in the specimen (3) ground by a 100 grit size diamond wheel (4) thickness of coating was approximately 0.002 in. |
| 22 | Aluminized | (1) mechanically polished as described above (2) specimen was aluminized in an argon atmosphere at 1350 F for 4 min and subsequently heated for 15 min at 1900 F to allow diffusion. |
| 23 | Screen | (1) mechanically polished as described above (2) nickel-plated copper screen of 40 mesh size placed between the gripping pads and the specimen. |
| 24 | Screen | Same as 23 only 80 mesh size screen used. |

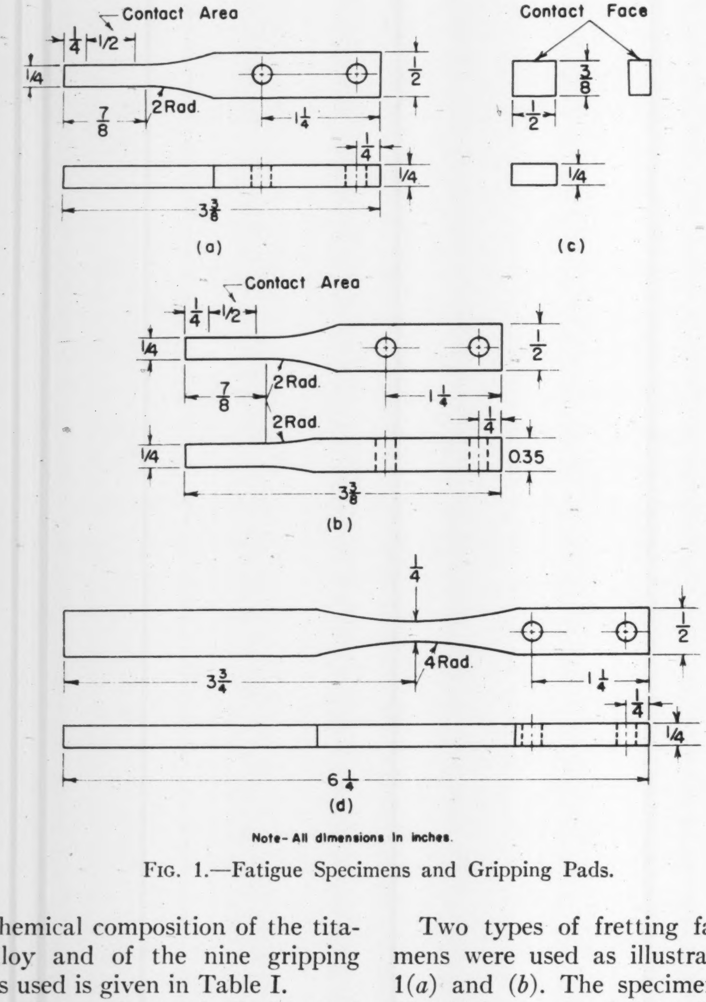
This study was undertaken to clarify the mechanism of fretting fatigue damage and to suggest possible remedies to the problem. The material used for all fatigue specimens was RC 130B titanium alloy. The influence of such variables as gripping pressure, gripping pad materials, and specimen surface treatment were studied. Magnesium, several aluminum alloys, copper, brass, titanium alloy, and steel of several hardnesses were used as gripping pad materials. Surface treatments of the titanium included oxidizing, shot peening, teflon coating, tungsten carbide plating, and aluminizing. To explore the influence of oxidation and debris in causing failure, some fretting fatigue tests were conducted in an atmosphere of dry argon, and in other experiments nickel-coated copper screens were employed as inserts or cushions between the gripping pads and the specimens.

## EXPERIMENTAL INVESTIGATION AND RESULTS

### *Materials and Specimens:*

All fretting specimens were made of titanium alloy RC 130B which was received as $\frac{1}{2}$ by $\frac{3}{8}$-in. rectangular rod The mechanical properties were determined to be:

Tensile Strength, 162,000 psi
Yield Strength (tensile), 149,700 psi
Elongation, 16.5 per cent (2-in. gage length)


Contact Area
1/4
1/2
1/4
2 Rad.
7/8
1 1/4
1/2
1/4
1/4
3 3/8
(a)
Contact Face
3/8
1/2
1/4
(c)
Contact Area
1/4
1/2
1/4
2 Rad.
7/8
2 Rad.
1 1/4
1/2
1/4
1/4
0.35
3 3/8
(b)
1/4
4 Rad.
3 3/4
1 1/4
1/2
1/4
1/4
6 1/4
(d)

Note- All dimensions in inches.

FIG. 1.—Fatigue Specimens and Gripping Pads.

The chemical composition of the titanium alloy and of the nine gripping materials used is given in Table I.

The mating surfaces of all the steel pads were finished by grinding, and all the non-ferrous pads were mechanically polished. Specimen surfaces as mechanically polished, oxidized, shot peened, tetrafluoroethylene resin (Teflon) coated, tungsten-carbide flame plated, and aluminized were tested. The preparation of these surfaces is described briefly in Table II. Also nickel-plated copper screens of 40 and 80 mesh sizes were used as inserts or cushions between the specimen and the SAE 4340 steel pads.

Two types of fretting fatigue specimens were used as illustrated in Figs. 1(*a*) and (*b*). The specimens shown in Fig. 1(*a*) were used throughout most of this investigation. In a few of the experiments, stresses nearly equal to the fatigue limit of the material were employed; therefore, the specimen shown in Fig. 1(*b*) was used to avoid an occasional failure away from the test section. The gripping pads are shown in Fig. 1(*c*), and the conventional fatigue specimen used to determine the fatigue limit for the material with a mechanically polished surface is shown in Fig. 1(*d*).

*Experimental Apparatus and Procedures:*

In Figs. 2(*a*) and (*b*), a schematic diagram and a photograph of the fretting fixture and experimental setup are shown. Specimen *A* was gripped between two pads, *B*, by a normal force applied through the steel beam, *C*, and by screws *K*. This normal force was determined from the deflection of the calibrated beam, *C*, using dial, *D*.

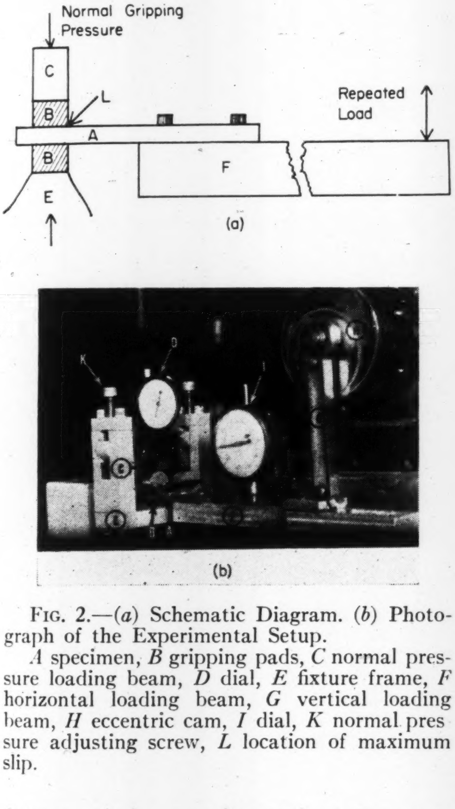


FIG. 2.—(*a*) Schematic Diagram. (*b*) Photograph of the Experimental Setup.

*A* specimen, *B* gripping pads, *C* normal pressure loading beam, *D* dial, *E* fixture frame, *F* horizontal loading beam, *G* vertical loading beam, *H* eccentric cam, *I* dial, *K* normal pressure adjusting screw, *L* location of maximum slip.

The specimen was rigidly connected to the horizontal loading arm *F*. The bending moment was applied by an eccentric cam *H* through the vertical loading crank *G*. Dial *I* was used to adjust and set the cam by reproducing the deflection corresponding to the desired stress as determined from a static load calibration.

Due to the repeated bending stress, slip occurred between the specimen and the gripping pads. In Fig. 2(*a*), *L* is the location of the maximum repeated slip. The slip was estimated to be of the order $10^{-4}$ to $10^{-5}$ in. (10). In all of the experiments, the mating surfaces were carefully cleaned with acetone prior to the assembly of the fixture.

A life of $5 \times 10^7$ cycles was defined as a run-out for all fretting fatigue experiments, and a life of $2 \times 10^7$ cycles was defined as a run-out for conventional fatigue experiments. Nine Krouse plate fatigue machines operating at 1800 rpm were used in this investigation.

*Experimental Results:*

Table III summarizes the fatigue results for all of the experimental conditions. A series number was assigned to each experimental condition and is listed in the first column of Table III. For convenience, it will be used to designate that condition throughout this report instead of repeatedly describing the detailed test conditions. The second column lists all of the gripping pad materials. The third and fourth columns give the diamond pyramid hardness numbers for gripping pads and specimens, respectively. Each hardness value in Table III represents the average of ten readings. The nominal gripping pressure and the surface preparations are listed; a more detailed description is included in Table II. The fretting fatigue strengths for each experimental condition are also listed. Since the term "fretting fatigue strength" will be used repeatedly, it will be called simply "fatigue strength" hereafter.

Figure 3 is arranged with decreasing "fatigue strength" toward the right, for

TABLE III.—SUMMARY OF RESULTS.

| Series | Gripping Material | Hardness of Gripping Pads, Diamond Pyramid Hardness Number | Hardness of Specimen, Diamond Pyramid Hardness Number | Gripping Pressure, psi | Surface Finish of Specimen | Fretting Fatigue Strength, psi | Remarks |
|---|---|---|---|---|---|---|---|
| No. 1...... | ... | ... | 354 | ... | Mechanically polished | 89 000 | Conventional fatigue test |
| No. 2...... | Magnesium | 39 | 354 | 15 000 | Mechanically polished | 85 000 | Adjusted fatigue limit |
| No. 3...... | Aluminum (1100-F) | 41 | 354 | 15 000 | Mechanically polished | 85 000 | Adjusted fatigue limit |
| No. 4...... | Aluminum (2011-T8) | 126 | 354 | 15 000 | Mechanically polished | 80 000 | ... |
| No. 5...... | Aluminum (7075-T6) | 194 | 354 | 15 000 | Mechanically polished | 38 000 | ... |
| No. 6...... | Copper | 96 | 354 | 15 000 | Mechanically polished | 72 000 | ... |
| No. 7...... | Brass (70-30) | 117 | 354 | 15 000 | Mechanically polished | 30 000 | ... |
| No. 8...... | Titanium (RC 130B) | 354 | 354 | 15 000 | Mechanically polished | 36 000 | ... |
| No. 9...... | Lapelloy | 207 | 354 | 40 000 | Mechanically polished | 24 000 | ... |
| No. 10...... | Steel SAE 4340 | 397 | 354 | 4 000 | Mechanically polished | 28 000 | ... |
| No. 11...... | Steel SAE 4340 | 397 | 354 | 22 000 | Mechanically polished | 21 000 | ... |
| No. 12...... | Steel SAE 4340 | 397 | 354 | 60 000 | Mechanically polished | 18 000 | ... |
| No 13...... | Steel SAE 4340 | 238 | 354 | 40 000 | Mechanically polished | 21 000 | ... |
| No. 14...... | Steel SAE 4340 | 397 | 354 | 40 000 | Mechanically polished | 22 000 | ... |
| No. 15...... | Steel SAE 4340 | 595 | 354 | 40 000 | Mechanically polished | 19 000 | ... |
| No. 16...... | Steel SAE 4340 | 238 | 354 | 40 000 | Mechanically polished | 21 000 | Argon atmosphere |
| No. 17...... | Steel SAE 4340 | 238 | ... | 15 000 | Oxidized | 50 000 | ... |
| No. 18...... | Aluminum (7075-T6) | 194 | ... | 15 000 | Shot peened | 55 000 | ... |
| No. 19...... | Steel SAE 4340 | 238 | ... | 40 000 | Shot peened | 48 000 | ... |
| No. 20...... | Steel SAE 4340 | 238 | ... | 15 000 | Teflon coated | 51 000 | ... |
| No. 21...... | Tungsten carbide<br>Plated lapelloy | 946 | 870 | 40 000 | Tungsten carbide plated | 21 000 | ... |
| No. 22...... | Aluminized lapelloy | ... | ... | 15 000 | Aluminized | 22 000 | ... |
| No. 23...... | Screen and steel SAE 4340 | ... | 354 | 40 000 | Mechanically polished | 44 000 | Nickel plated copper screen[a] |
| No. 24...... | Screen and steel SAE 4340 | ... | 354 | 40 000 | Mechanically polished | 34 000 | Nickel plated copper screen[b] |

[a] Nickel plated copper screens of mesh size 40 were used as cushions or inserts between the gripping pads and the specimens.

[b] Same as above, except screens of mesh size 80 were used.

all of the different experimental conditions. It is evident that the "fatigue strengths" for essentially identical specimens, that is, the same mechanically polished specimens, vary over a considerable range depending upon the gripping pads used. The highest "fatigue strengths" were approximately equal to the fatigue limit of the material and occurred with the very soft gripping pad materials. The lowest "fatigue strengths" were approximately 0.20 of the fatigue limit of the material and occurred when hard gripping pads were employed.

The "fatigue strength" of the mechanically polished specimens can be divided into two groups. Values for the high "fatigue strength" group are near the ordinary fatigue limit of the material, while those for the low "fatigue strength" group are approximately one-fourth of the fatigue limit. The *S-N* curves within each group are very similar; therefore only representative curves are shown, except for series 6 and 7, where copper and brass pads were used. Figure 4(*a*) shows the *S-N* curve of mechanically polished specimens without fretting. The *S-N* curves in Fig. 4(*b*) and (*c*) were obtained using aluminum 2011 pads and SAE 4340 steel pads and are representative of the high and low "fatigue strength" groups, respectively. For copper and brass pads, the specimens had unusually long lives, and these two curves were shifted to the extreme right as shown in Fig. 4(*d*) and (*e*). Whether the "knee" in these curves actually exists is not known. The long time that would be required to establish a "knee"

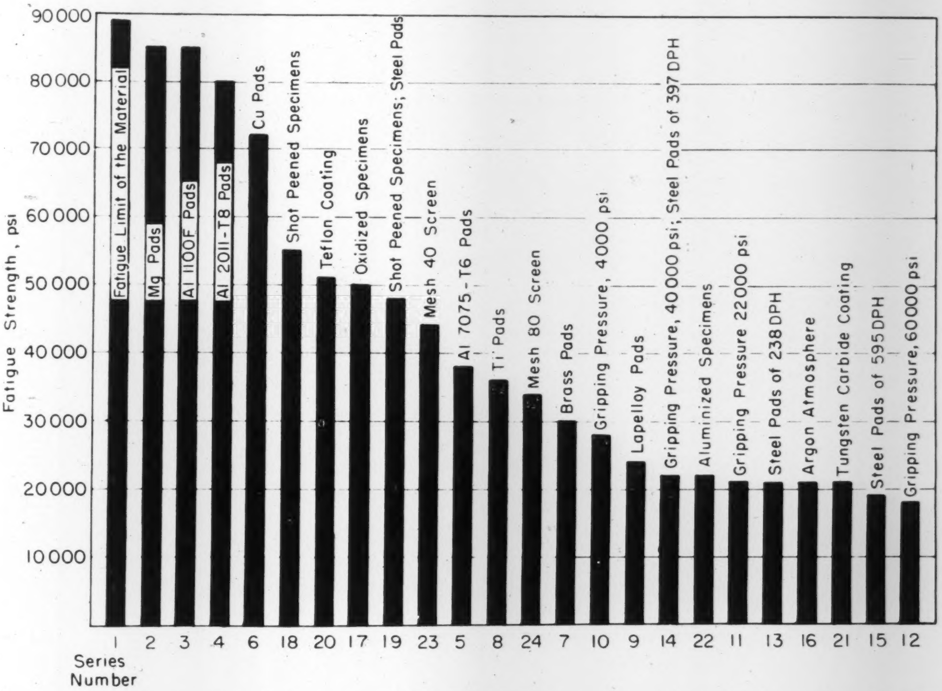


Fig. 3.—Fretting Fatigue Strength of RC 130B Titanium Alloy Under Various Experimental Conditions.

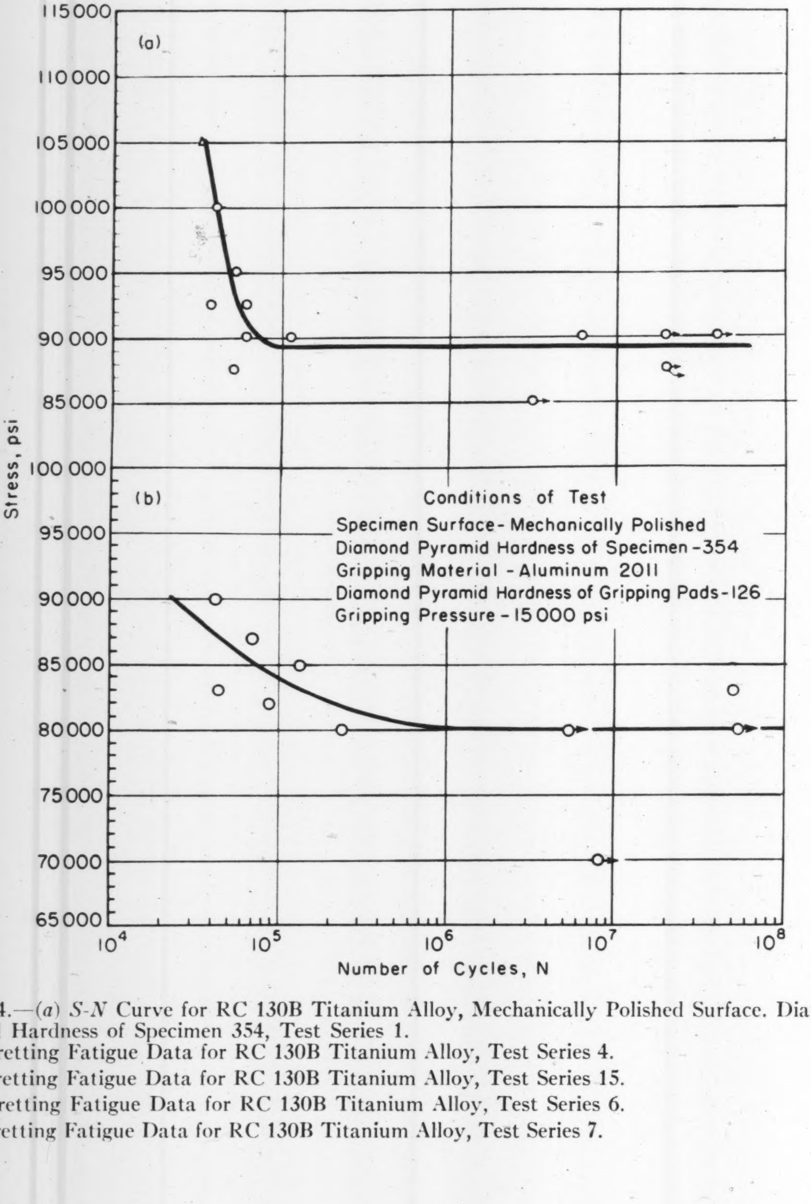


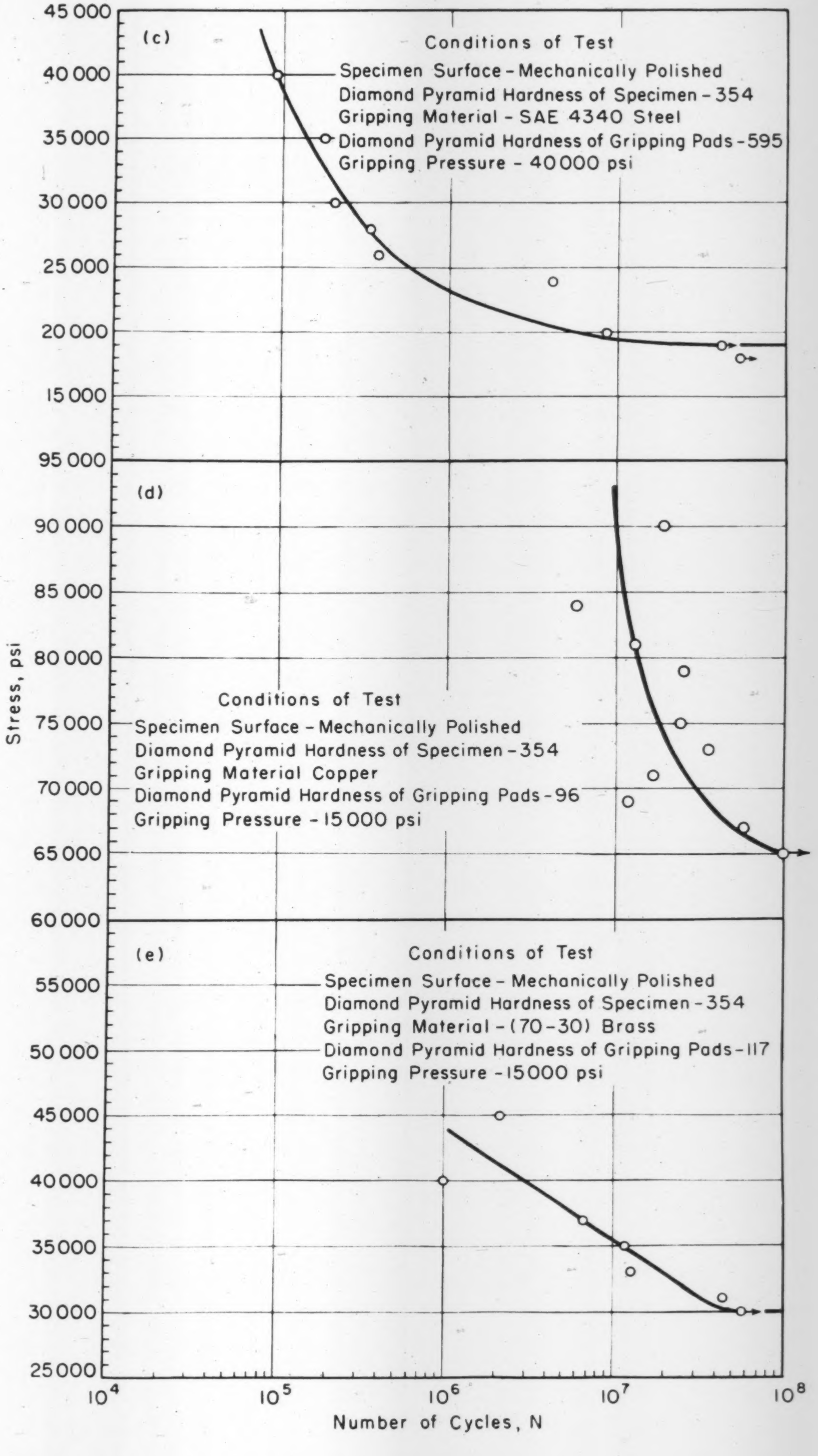
FIG. 4.—(*a*) *S-N* Curve for RC 130B Titanium Alloy, Mechanically Polished Surface. Diamond Pyramid Hardness of Specimen 354, Test Series 1.

(*b*) Fretting Fatigue Data for RC 130B Titanium Alloy, Test Series 4.

(*c*) Fretting Fatigue Data for RC 130B Titanium Alloy, Test Series 15.

(*d*) Fretting Fatigue Data for RC 130B Titanium Alloy, Test Series 6.

(*e*) Fretting Fatigue Data for RC 130B Titanium Alloy, Test Series 7.

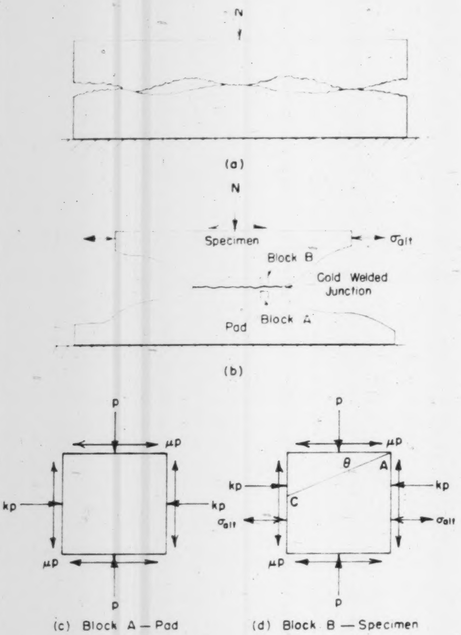

(c)
Conditions of Test
Specimen Surface - Mechanically Polished
Diamond Pyramid Hardness of Specimen - 354
Gripping Material - SAE 4340 Steel
Diamond Pyramid Hardness of Gripping Pads - 595
Gripping Pressure - 40000 psi
45 000
40 000
35 000
30 000
25 000
20 000
15 000
(d)
Conditions of Test
Specimen Surface - Mechanically Polished
Diamond Pyramid Hardness of Specimen - 354
Gripping Material Copper
Diamond Pyramid Hardness of Gripping Pads - 96
Gripping Pressure - 15 000 psi
95 000
90 000
85 000
80 000
75 000
70 000
65 000
60 000
(e)
Conditions of Test
Specimen Surface - Mechanically Polished
Diamond Pyramid Hardness of Specimen - 354
Gripping Material - (70-30) Brass
Diamond Pyramid Hardness of Gripping Pads - 117
Gripping Pressure - 15000 psi
55000
50000
45000
40000
35000
30000
25000
Stress, psi
$10^4$
$10^5$
$10^6$
$10^7$
$10^8$
Number of Cycles, N

in the range of $5 \times 10^7$ cycles was prohibitive.

## DISCUSSION OF RESULTS

### *Model of Mechanical Mechanism of Fretting Fatigue Crack Initiation:*

The modern theory of friction and wear is based on the fact that when two metal surfaces are pressed into contact, only the high asperities[3] of these two surfaces actually contact (**11**). These asperities yield and cold weld together as shown schematically in Figs. 5(*a*) and (*b*). When the two surfaces undergo a relative lateral displacement, these welded asperities break either at the interface or within the asperities. The failure may simulate that of static fracture if the relative velocity is low and the displacement is large; or if the displacement is repeated and small, thousands of cycles of loading may be necessary to produce progressive fracture (fatigue) of the asperities.



(c) Block A — Pad (d) Block B — Specimen

FIG. 5.—Contacting Asperities and Associated Stresses.

If the strengths of the two mating metals are different, the weaker surface will be damaged severely and the stronger surface may or may not be damaged depending on whether the strongest asperity of the weaker surface is stronger or weaker than the weakest asperity of the stronger surface.

With the present experimental setup, welded asperities near the edge *L*, as shown in Fig. 2(*a*), will break with one or several cycles of loading due to the relatively large displacement. If the failure takes place through the specimen asperities, the surface will be roughened and the fatigue strength will be reduced, but not to the extent of 0.2 of the fatigue limit of the material as reported in the previous section.

At locations away from the edge of the grip, the relative longitudinal displacement, which induces the repeated frictional shear stress between these two surfaces, decreases. It is in this region, a short distance inside of the grip, that fatigue damage and progressive fracture have been observed to occur (**6**). This repeated frictional shear stress together with the repeated bending stress at a particular location initiates fatigue cracks and causes fatigue damage. The welded asperities apparently develop small cracks only after thousands of cycles of loading have been applied.

By considering the fully plastic condition of the weaker asperities, governed by the coefficient of friction and the yield pressure of the weaker of the two mating metals, the maximum repeated frictional shear stress that can be developed at the interface may be calculated. If the combined influence of the frictional shear stress and the bending stress is larger than the fatigue limit of the specimen material, fatigue cracks will be initiated. In the analysis of the stresses on the specimen and the gripping pad asperities that follows, it will be shown that the "fatigue strength" is related to the fatigue limit of the specimen material, the hardness of the gripping pad, and the coefficient of friction.

---

[3] Minute high spots, points of contact when two metal surfaces are pressed together.

While it is not a necessary requirement, it will be assumed for convenience of presentation that the yield strength of the gripping pad material is lower than that of the specimen material. The following symbols will be used in the analysis:

| | |
|---|---|
| $\tau_{\max}$ | maximum shear stress in the asperities of gripping pad, psi, |
| $p$ | normal pressure on the asperities, psi, |
| $\mu$ | coefficient of friction, |
| $P$ | yield pressure of the weaker metal in the fully plastic state, psi, |
| $\sigma_y$ | yield strength of the weaker material, psi, |
| $H$ | diamond pyramid hardness number of gripping pads, kg per sq mm, |
| $\tau_{\text{alt}}$ | alternating shear stress in specimen due to alternating bending stress and frictional shear stress, psi, and |
| $\sigma_{\text{alt}}$ | alternating bending stress in specimen, psi. |

*Gripping Pad.*—Figure 5(*c*) shows the stresses acting on an infinitesimal block of the material of the gripping pads at the interface. The maximum shear stress on the block is:

$$\tau_{\max} = p\sqrt{\left(\frac{1-k}{2}\right)^2 + \mu^2} \quad \ldots\ldots (1)$$

From the two-dimensional solution of an ideal plastic metal loaded by a flat punch without friction, $k$ is equal to $\frac{\pi}{2+\pi}$ [4] (12). For the fully plastic condition of the asperities, $\tau_{\max}$ is equal to the shearing yield strength of the gripping pad material, that is, $\tau_{\max} = \sigma_y/2$. Studies of plastic indentation of metals give the following relationships (12):

$$P = 3\sigma_y$$

and

$$P = 1420 \times \frac{H}{0.927}$$

where $\sigma_y = \frac{H}{0.927}$ for a 136 deg diamond pyramid indenter and 1420 is the conversion factor for Kg per sq mm to psi. Using these expressions and Eq 1, the following relation between $p$, $H$, and $\mu$ is obtained:

$$p^2 = 2.61 \times 10^5 \frac{H^2}{0.151 + 4\mu^2} \quad \ldots\ldots (2)$$

where $p$ is the maximum normal pressure that the pad asperities can sustain in a fully plastic state, when a frictional shear stress $\mu p$ and normal pressures $p$ and $kp$ are superimposed.

*Specimen.*—Figure 5(*d*) shows the stresses acting on an infinitesimal block of material of the specimen at the interface. On plane $AC$, when $\sigma_{\text{alt}}$ is tensile, the shear stress, $\tau_1$, is equal to

$$\tau_1 = \tfrac{1}{2}(1-k)p \sin 2\theta - \mu p \cos 2\theta + \tfrac{1}{2}\sigma_{\text{alt}} \sin 2\theta$$

When $\sigma_{\text{alt}}$ is compressive, the shear stress on plane $AC$, $\tau_2$, is equal to

$$\tau_2 = \tfrac{1}{2}(1-k)p \sin 2\theta + \mu p \cos 2\theta - \tfrac{1}{2}\sigma_{\text{alt}} \sin 2\theta$$

The amplitude of the repeated shear stress $\tau_{\text{alt}}$, is equal to

$$\tau_{\text{alt}} = \tfrac{1}{2}(\tau_1 - \tau_2)$$

$$\tau_{\text{alt}} = \tfrac{1}{2}\sigma_{\text{alt}} \sin 2\theta - \mu p \cos 2\theta$$

The maxima of $\tau_{\text{alt}}$ with respect to $\theta$ were determined to be

$$\tau_{\text{alt}} = \tfrac{1}{2}\sqrt{4\mu^2 p^2 + \sigma_{\text{alt}}^2} \quad \ldots\ldots (3)$$

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[4] To use the value of $k$ for the solution of a flat punch without friction introduces error, but this analysis shows that the error is negligible, if $\mu$ is equal to or larger than 0.5.

By setting $\tau_{\text{alt}}$ equal to the shearing fatigue limit ($\frac{1}{2}$ of bending fatigue limit) of the specimen material and using Eq 2 for the value of $p$, Eq 3 can be solved for $\sigma_{\text{alt}}$, the allowable bending stress, in terms of $\tau_{\text{alt}}$, $H$, and $\mu$, to be:

$$\sigma_{\text{alt}} = \sqrt{4\tau_{\text{alt}}^2 - 1.04 \times 10^6 \frac{\mu^2 H^2}{0.151 + 4\mu^2}} \quad \ldots (4)$$

Equation 4 gives the limiting values of $\sigma_{\text{alt}}$ for preventing fatigue crack initiation and indicates that, for a given gripping pad hardness, the "fatigue strength," $\sigma_{\text{alt}}$, increases with a decrease in the coefficient of friction or hardness. As the coefficient of friction or hardness approaches zero, the "fatigue strength" approaches the fatigue limit of the specimen material.

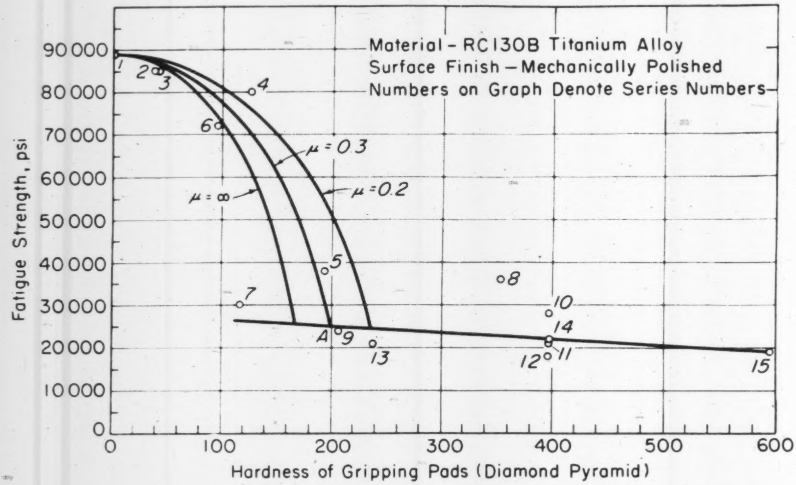

FIG. 6. Influence of Hardness of Gripping Pads on Fretting Fatigue Strength.

*Gripping Pad Hardness:*

For particular values of $\mu$ and $H$, fatigue cracks will be initiated if the applied repeated bending stress is higher than $\sigma_{\text{alt}}$ given by Eq 4. If $\sigma_{\text{alt}}$ is lower than the value given by Eq 4, no fatigue cracks will be initiated and fatigue failure will not occur. Based on Eq 4, three curves were drawn in Fig. 6 corresponding to values of $\mu$ of $\infty$, 0.3, and 0.2, and using $\tau_{\text{alt}} = \frac{1}{2}$ 89,000 = 44,500 psi. For a range of values of $\mu$ from 1 to $\infty$ the curves are nearly identical and result in a shift in the horizontal intercept (hardness) of the curves of only 2 per cent. As the value of $\mu$ decreases from 0.2 to 0, the horizontal intercept moves rapidly to the right. However, the relation becomes less reliable for a low coefficient of friction due to the error introduced by the value of $k$ in Eq 1. In Fig. 6, the curve for $\mu$ equal to $\infty$ can be used for all values of $\mu$ that are greater than 1. For most practical applications, the range of coefficients of friction is from 0.2 to 3. Thus, the band bounded by curves for $\mu$ equal to 0.2 and $\infty$ indicates the general relationship between the gripping pad hardness and the "fatigue strength." The most rapid drop in the "fatigue strength" occurs within the critical hardness range of 100 to 230 diamond pyramid hardness.

Figure 6 shows that, for any given value of $\sigma_{\text{alt}}$ and $\mu$, there is an upper limiting value of hardness, $H$, to avoid fatigue crack initiation. If $H$ is greater than this limit, fatigue cracks will be initiated. Once a crack is initiated, it may propagate into the specimen. As the depth of the crack increases, the amplitude of the repeated frictional shear stress, $\mu p$, decreases rapidly and approaches $\mu N$, or a value even lower than $\mu N$, where $N$ is the gripping pressure.[5] Therefore, the repeated bending stress, $\sigma_{\text{alt}}$, becomes the primary component of repeated stress which propagates the crack. The fretting fatigue member will not fracture if at any depth below the surface the resultant of the repeated bending stress and the surface-friction induced shear stress is lower than the stress necessary to propagate the crack. In other words, if the hardness of the gripping pad is higher than the limiting value prescribed in Fig. 6, fatigue cracks will be initiated at the surface and the "fatigue strength" will be determined by the bending stress at which the fatigue cracks cease to propagate.

As the hardness of gripping pads increases, the actual contact area between pad and specimen decreases and the frictional shear stress, $\mu p$, becomes larger. Consequently, at small depths beneath the surface, the shear stress will be greater, since the surface frictional shear stress, $\mu p$, is greater. Obviously this effect is rather small. However, the alternating bending stress that is necessary to propagate a crack decreases slightly with an increase of the hardness of the gripping pads, that is, the "fatigue strength" decreases slightly with an increase in gripping pad hardness. Based on the experimental data, a straight line was drawn through the data in Fig. 6. For $\mu = 0.3$, this straight line intersects the curve governing crack initiation, Eq 4, at point $A$. If the gripping pad hardness is lower than the abscissa of $A$, the "fatigue strength" is governed by crack initiation. If the gripping pad hardness is higher than the abscissa of $A$, the "fatigue strength" is governed by crack propagation.

The experimental results for mechanically polished specimens with gripping pads of various hardnesses are also plotted in Fig. 6. Considering the normal scatter of fatigue data, the variation of gripping pressure employed in the different test series, and the simplifications and assumptions made in the analysis, the experimental data shown in Fig. 6 confirm the general pattern of the analytical curves very well. The data for series 4, 7, and 8 show the greatest deviations. Whether these deviations are due to unusual scatter of the data or due to the simplifications made in the analysis[6] is not known. It does appear, however, that the relationship between "fatigue strength," that is either crack initiation or propagation, and hardness is adequately verified by the data.

It is interesting to note that in test

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[5] If the frictional shear stress at all the contacted asperities, longitudinally across the contact area was $\mu p$, the shear stress beneath the surface would approach $\mu N$. If the frictional shear stress at the asperities near the edge $L$, Fig. 2($a$), is $\mu p$, but decreases to zero toward the left edge; the shear stress beneath the surface will approach a value lower than $\mu N$.

[6] The simplifications and assumptions made in the analysis are as follows:

($a$) The ratio between the yield pressure and the yield strength was assumed to be 3. This ratio can vary from 2.6 to 3.3 depending upon the state of cold work of the asperities (**12**).

($b$) A super-imposed static compressive stress does not affect the fatigue strength of the material. The available evidence generally supports this assumption (**13**), however, the state and magnitude of the super-imposed static compressive stress encountered in the fretting specimens was somewhat different from any that have been investigated.

($c$) Any influence of the debris particles was neglected. These particles may possibly act as a solid lubricant or as an abrasive. The alloying effect (**14, 15**) between the two mating surfaces and the change of the surface hardness of the specimen and the gripping pads due to cold working during repeated sliding was neglected.

series 6 and 7, in which the data in Fig. 6 lie to the left and below the solid curves, copper or copper-base alloy gripping pads were used. By comparison, series 4 and 5, which employed aluminum alloy pads, showed higher "fatigue strength" at slightly higher hardnesses. This observation correlates with a previous finding using similar pad materials and steel specimens (**16**).

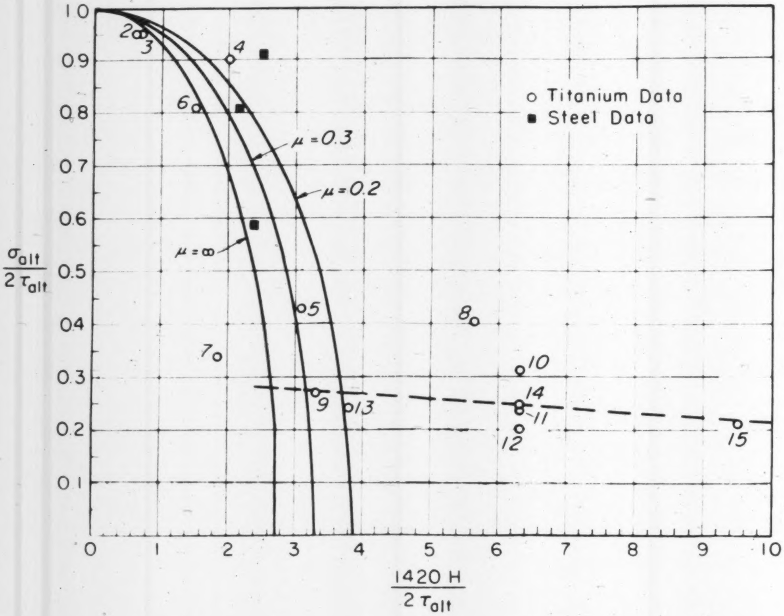

FIG. 7.—Dimensionless Diagram Showing Fretting Fatigue Strength as Influenced by the Fatigue Limit of the Material, the Hardness of the Gripping Pads, and the Coefficient of Friction.

Equation 4 may be rearranged in dimensionless form to give:

$$\left[\frac{1420H}{2\tau_{alt}}\right]^2 = 1.933\left[1 - \left(\frac{\sigma_{alt}}{2\tau_{alt}}\right)^2\right]\left[\frac{0.151}{\mu^2} + 4\right] \quad \text{..(5)}$$

If the dimensionless quantities, $\frac{\sigma_{alt}}{2\tau_{alt}}$ and $\frac{1420\ H}{2\tau_{alt}}$, are plotted as ordinate and abscissa respectively for various values of $\mu$, as shown in Fig. 7, the curves based on Eq 5 are applicable for other materials, independent of the value of $\tau_{alt}$. Results for steel specimens with low hardness gripping pads (**16**) were found to fit the dimensionless plot, as shown in Fig. 7 by the solid square points. However, the value of the ratio $\frac{\sigma_{alt}}{2\tau_{alt}}$, for which cracks cease to propagate, that is the "fatigue strength" for $\frac{1420\ H}{2\tau_{alt}} > 4.0$, appears to depend upon the specimen material. More work in this area is desirable.

If a linear relationship is assumed between $H$ and $\sigma_{alt}$ for fatigue crack propagation, the extrapolation of the straight line of Fig. 6 to $H = 0$ gives the stress that is necessary to propagate a crack in the absence of fretting. The value was approximately 30,000 psi.

*Gripping Pressure:*

It is obvious from the above analysis that the frictional shear stress, $\mu p$, is independent of gripping pressure, because $p$ depends only on the hardness of gripping pad, $H$, and the coefficient of friction, $\mu$. Since the initiation of fatigue cracks depends on $\mu p$ and $\sigma_{\text{alt}}$ only, it is also independent of gripping pressure. However, the surface-friction induced shear stress at a small distance beneath the surface of the specimen is related to $\mu N$. This shear stress increases with the gripping pressure. Since the propagation of a crack depends on the resultant of the surface friction induced shear stress and the bending stress, $\sigma_{\text{alt}}$, an increase in the surface-friction induced shear stress decreases the bending stress, $\sigma_{\text{alt}}$, that is necessary to propagate a crack. However, the surface-friction induced shear stress component is small, and an increase in gripping pressure should decrease the "fatigue strength" only slightly. In series 10, 11, 12, and 14, gripping pads of SAE 4340 steel with a diamond pyramid hardness of 397 were employed. Nominal gripping pressures ranging from a minimum of 4000 psi to a maximum of 60,000 psi were used. The experimental results in Fig. 8 indicate the decrease in "fatigue strength" due to an increase in gripping pressure is very small. If a linear relationship between gripping pressure and "fatigue strength" is assumed, extrapolation to zero pressure gives the stress that was necessary to propagate the fatigue cracks in the absence of fretting. Extrapolation gave a value of approximately 30,000 psi, which is about the same as the value obtained by extrapolation of the straight line in Fig. 6 to zero hardness.

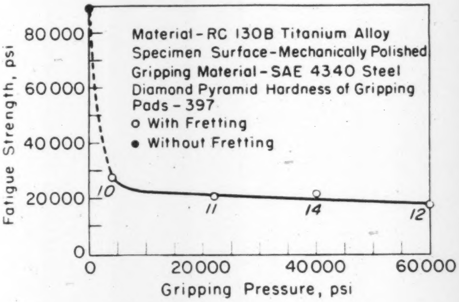

FIG. 8.—Influence of Gripping Pressure on Fretting Fatigue Strength.

*Oxidation:*

A comparison of the results of test series 13, 16, and 17 indicates the influence of an oxide layer on the surface of the specimen. Series 16, in which the experimental conditions were the same as in series 13 except that an argon gas atmosphere was employed, gave a "fatigue strength" of 21,000 psi, the same as that of series 13 which was run in air. This result agrees with the interpretation of the proposed hypothesis of fatigue damage at the cold-welded asperities by the repeated bending stress and the superimposed repeated frictional shear stress.

In series 17, two batches of specimens were deliberately oxidized. Specimens of batch No. 1 were immersed in hydrogen peroxide for 2 hr, while the specimens of batch No. 2 were immersed for 16 hr. The "fatigue strength" of batch 1 specimens, 50,000 psi, was approximately 240 per cent of that of series 13, in which comparable experimental conditions were used except that the surface was mechanically polished. Batch No. 2, which was oxidized for a longer time, exhibited a lower (but undetermined) "fatigue strength" than batch No. 1. The increased strength of the oxidized specimens compared to mechanically polished specimens is presumably due to the thicker protective oxide layer. The oxide layer prevents clean metal contact and solid phase welding and reduces the coefficient of friction. A lower coefficient of friction reduces the repeated frictional shear stress, which is the main component of the repeated stress on the specimen asperities. Consequently, the protective functioning of the oxide layer apparently depends on the properties of the oxide, that is, the strength of the oxide, the bond between the oxidized layer and the base metal, preferential attack of the surface, and the thickness and volumetric change of the layer during the oxidizing process.

The results of test series 16 and 17 suggest that the occurrence of normal oxidation during the progress of the experiment is an insignificant part of the surface damage when fretting is measured in terms of fatigue strength.

### *Special Surface Preparations:*

These surface preparations include shot peening, teflon coating, tungsten carbide plating, aluminizing, and the use of nickel-plated copper screens as inserts or cushions between the gripping pads and the specimen.

*Shot Peening.*—The specimens of series 18 and 19 were shot peened, with steel shot size P39 and P19 respectively. The gripping pad materials were aluminum 7075-T6 for series 18 and SAE 4340 steel for series 19. The "fatigue strength" was improved to 55,000 and 48,000 psi respectively. The "knee" of the $S$-$N$ curve did not appear in series 18, within the runout life of $5 \times 10^7$ cycles.

The shot peening process cold worked the specimen surface and apparently raised the stress necessary to initiate as well as to propagate cracks. It is presumed that cracks were initiated by the proposed mechanism and the "fatigue strength" of series 19 was the stress at which the cracks ceased to propagate.

The long life of these specimens, particularly series 18, may be attributable to very slow propagation of the cracks within the cold-worked layer.

*Teflon Coating.*—Series 20, the Teflon-coated specimens, gave a "fatigue strength" of 51,000 psi. Teflon has a low coefficient of friction and exhibits a strong bond with the base material. Initially, at least, it serves as a buffer between the two metal surfaces and prevents metal to metal contact. However, Teflon will be gradually worn away (**17**). When metal to metal contact eventually occurs, the increased repeated frictional shear stress may reduce the "fatigue strength." Therefore, the protection afforded by a Teflon coating appears to be dependent upon the thickness of the coating and the desired life of the part.

Both shot peening and Teflon coating appear to offer practical remedies that reduce the fretting damage.

*Tungsten Carbide Flame Plating.*—Series 21, the tungsten carbide flame plated specimens, gave a strength of 21,000 psi. The fatigue fracture was perpendicular to the surface and no tendency for separation of the plating was noticed. To determine whether this reduced strength was due to a metallurgical change of the material or surface damage during the processing requires more exploratory study. Another possible explanation is that the tensile strength of the coating is low, and cracks formed within the coating serve as notches in the specimen (**18**).

*Aluminizing.*—In view of the high "fatigue strengths" of series 3 and 4, it was thought that if a layer of aluminum were applied to the specimen and the lapelloy pads, the lapelloy-titanium contact could be prevented and the "fatigue strength" increased. Therefore, test series 22 was conducted using aluminized specimens. The estimated thickness of the aluminum layer was approximately 0.003 in. and a "fatigue strength" of 22,000 psi was obtained.

This reduced "fatigue strength" is believed to be due to the fact that the coating was too thin and was rapidly worn away. The thickness of the aluminized and the oxide layers were very similar, but they served different functions. The oxide layer was harder, adhered to the surface, and reduced the coefficient of friction, thereby reducing the repeated frictional shearing stress.

The aluminum layer served as a buffer separating the gripping pads and the specimen. However, the soft thin layer was apparently rapidly worn off; consequently, no beneficial increase of "fatigue strength" was obtained. The alloying effect between the aluminum and the titanium and the high temperature during the aluminizing process also may have adversely affected the fatigue properties.

*Screens.*—The nickel-plated copper screens of mesh sizes 40 and 80 used in series 23 and 24 resulted in increased "fatigue strengths" of 44,000 and 34,000 psi respectively. The reason for this improvement is not well understood since the screen may function in several ways. The increased "fatigue strength" may be due to the low hardness of the nickel and copper, or due to the fact that the relative lateral displacement between the gripping pad and the specimen was partially absorbed by the screen. In the latter case, the relative displacement between the screen and the specimen asperities may have been reduced to the point that an increased bending stress was necessary to cause fatigue failure.

If debris particles are at all significant the open grid of the screen certainly offers ample space to remove the particles from critical regions.

### *X-ray Study of Debris:*

The fretting debris from test series 2, 3, 6, 7, and 9 were analyzed by X-ray to determine its composition. For test series 2, 3, and 6, which gave "fatigue strengths" ranging from 85,000 psi to 72,000 psi, no titanium or titanium alloy was detected in the debris. For series 7 and 9, which gave low "fatigue strengths" of 30,000 psi and 24,000 psi, respectively, the X-ray results indicated that the debris includes titanium alloy. This suggests that when a high "fatigue strength" was obtained, the gripping pads did not damage the specimen. Conversely, if a low "fatigue strength" was obtained, the X-ray analysis indicated that the specimen surface was damaged, that is, titanium in some form was found in the debris. These results are in agreement with the proposed mechanism of fatigue crack initiation discussed previously.

### *Conclusions:*

The results of this investigation appear to justify the following conclusions:

1. Severe fretting damage reduces the fatigue strength of RC 130B titanium alloy to as low as 0.2 of the ordinary fatigue limit of the alloy.

2. The mechanism responsible for fretting fatigue damage appears to be the repeated frictional shear stress on the contacted asperities of the specimen surface. A mathematical expression was derived which relates the fretting fatigue strength to the fatigue limit of the specimen material, the hardness of the gripping pad, and the coefficient of friction. The experimental data seem to be in good agreement with the analytical expression.

3. To reduce the severity of fretting damage and increase the fretting fatigue strength, the analysis suggests the following remedies: (*a*) reduce the gripping pad hardness below the critical hardness range of 100 to 230 diamond pyramid hardness (*b*) reduce the coefficient of friction below 0.1, (*c*) increase the stress that is necessary to propagate a fatigue crack, and (*d*) prevent metal to metal contact.

4. Fretting caused by gripping pads of hardness above the critical range, reduces the fretting fatigue strength to

the range of 0.2 to 0.4 of the fatigue limit of the titanium alloy specimens. Gripping pads of low hardness, below the critical range, give fretting fatigue strengths above 0.8 of the fatigue limit of the specimen.

5. Special surface treatments employing either an oxidized layer or teflon coating increase the fretting fatigue strength to approximately 0.56 of the fatigue limit of the alloy. The oxidized layer and the Teflon coating each prevent metal to metal contact and the coefficient of friction.

6. Shot peening improves the fretting fatigue strength to 0.5 to 0.6 of the ordinary fatigue limit of the titanium alloy.

7. Exclusion of an oxygen (or air) atmosphere did not result in an improved fretting fatigue strength.

8. The two surface treatments tungsten carbide plating and aluminizing resulted in no increase of the fretting fatigue strength.

9. Using nickel-plated copper screens as inserts or cushions between the gripping pad and the specimen, increased the fretting fatigue strength slightly.

*Acknowledgment:*

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## REFERENCES

(1) Douglas Godfrey, "Investigation of Fretting Corrosion by Microscopic Observation," *Technical Note No. 2039*, Nat. Advisory Comm. Aeronautics, Feb., 1950.

(2) B. W. Sakmann, and B. G. Rightmire, "An Investigation of Fretting Corrosion Under Several Conditions of Oxidation," *Technical Note No. 1492*, Nat. Advisory Comm. Aeronautics, June, 1948.

(3) M. Fink, "Wear Oxidation, A New Component of Wear," *Transactions*, Am. Soc. Steel Treating, Vol. 18 (1936).

(4) I-Ming Feng, and B. G. Rightmire, "The Mechanism of Fretting," *Lubrication Engineering*, June, 1953.

(5) H. H. Uhlig, I-Ming Feng, W. D. Tierney, and A. McClellan, "A Fundamental Investigation of Fretting Corrosion," *Technical Note No. 3029*, Nat. Advisory Comm. Aeronautics, Dec., 1953.

(6) O. J. Horger, "Influence of Fretting Corrosion on the Fatigue Strength of Fitted Members," Symposium on Fretting Corrosion, Am. Soc. Testing Mats. p. 40 (1953). (Issued as separate publication *ASTM STP No. 144.*)

(7) O. J. Horger and J. L. Maulbetsch, "Increasing the Fatigue Strength of Press-Fitted Axle Assemblies by Surface Rolling," *Journal of Applied Mechanics*, Vol. 3, No. 3, Sept., 1936.

(8) A. Thum and F. Wunderlich, "Dauerbiegefestigkeit von Konstrucktionsteilen an Einspannungen, Nabensitzen und ahnlichen Kraftangriftstellen," Mitteilungen der Materialprufungsanstalt an der Technischen Hochschule Darmstadt, Heft 5, Zeitschrift des Vereines deutscher Ingenieure—Verlag, Berlin (Germany) (1934).

(9) R. E. Peterson and A. M. Wahl, Fatigue of Shafts at Fitted Members, With a Related Photoelastic Analysis," *Journal of Applied Mechanics*, March, 1935.

(10) H. W. Liu, H. T. Corten, and G. M. Sinclair, "Fretting Fatigue Strength of

Titanium Alloy RC 130B," *Report No. 107*, Department of Theoretical and Applied Mechanics, Univ. of Illinois, Urbana, Ill., Sept., 1956.

(11) F. P. Bowden and D. Tabor, "The Friction and Lubrication of Solids," Oxford University Press, New York, N. Y. (1950).

(12) D. Tabor, "The Hardness of Metals," Oxford University Press, London, 1951.

(13) J. O. Smith, "The Effect of Range of Stress on the Fatigue Strength of Metals," *Engineering Experiment Station Bulletin*, Series No. 334, Univ. of Illinois, Urbana, Ill. (1942).

(14) E. S. Machlin, "Research Points Way to New Methods of Preventing Galling and Seizing," *Iron Age*, Feb. 10, 1955.

(15) L. F. Coffin, Jr., "Study of the Sliding of Metals, with Particular Reference to Atmosphere," *Lubrication Engineering*, Jan.-Feb., 1956.

(16) H. T. Corten, "Factors Influencing Fretting Fatigue Strength," Department of Theoretical and Applied Mechanics, Univ. of Illinois, Urbana, Ill., (1955).

(17) W. P. Mason and S. D. White, "New Techniques for Measuring Forces and Wear in Telephone Switching Apparatus," *Bell System Technical Journal*, Vol. 31, p. 469 (1952).

(18) G. W. Form and W. M. Baldwin, "The Effect of Brittle Skins on the Ductility of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 645 (1956).

# DISCUSSION

MR. L. F. COFFIN, JR.[1]—This is a particularly interesting paper. The fact that the hardness of the materials in contact plays such an important role is quite significant. One question arises in the analysis as to whether one should not distinguish between the case where the pad material is harder or softer than the base material. For example, when the pad is the softer, then a direct relationship would exist between hardness and normal pressure. On the other hand, when the pad material is harder than the titanium, that relationship no longer holds because, according to the Bowden concept, all one does then is to continue to cause the titanium to yield with no further increase in pressure. This assumes that all one does is to bring more asperities in contact. I wonder whether if this fact were introduced in your analysis you could not predict analytically the dashed line shown in Fig. 7.

MR. H. W. LIU (*author*).—If the gripping pad is harder than the specimen material, cracks will be initiated and may be propagated depending on the magnitude of the bending stress. The scattering on the right part of Fig. 7 is due to factors other than hardness, such as different gripping pressures, etc.

(*Authors' closure*).—The authors wish to thank Mr. Coffin for his remarks. With regard to the analysis presented in the paper dealing with crack initiation, it is unnecessary to distinguish between the case where the pad material is harder or softer than the base material (specimen material). However, it is necessary to distinguish whether the frictional shear stress, which is limited by the hardness of either pad or base material, combined with the bending stress is enough to initiate a crack. The critical limiting hardness of the pad in order to avoid crack initiation in the specimen is much lower than the hardness of the base (specimen) material. Therefore, in the analysis of *crack initiation*, the case where the pad material is harder than the base material, is entirely outside of the range of interest.

The dashed line in Fig. 7 is believed to be associated with the smallest stress that will cause crack propagation. In order to predict the dashed line analytically, additional information is needed including the relationship between the depth of the crack and the stress necessary to further propagate the crack, plus the state of stress at the tip of the crack. Since this information was lacking, the dashed line in Fig. 7 was introduced empirically to fit the data.

It may be proper to question whether or not the dashed line in Fig. 7 should be straight and continuous at the point where the pad and specimen are of the same hardness; however from the available data the dashed line appears to adequately represent the data.

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# CRACKING OF NOTCH FATIGUE SPECIMENS*

By M. S. Hunter[1] and Wm. G. Fricke, Jr.[1]

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## Synopsis

The initiation and surface propagation of fatigue cracks within a series of rotating-beam fatigue specimens were observed at high magnifications. Specimens contained notches ranging in severity from very mild to extremely severe. The theoretical stress at the root of the notch largely controlled the number of cycles at which a crack formed and the rate of propagation of the crack along the notch, at least when the crack was small. Provided the theoretical stress at the notch root exceeded a critical value roughly identifiable with the endurance limit of smooth specimens, cracks formed even though the test stress was insufficient to cause complete failure. However, cracks formed at stresses below the notch endurance limit did not individually grow to large size.

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The complex design of modern structures necessarily involves notches, holes, and changes of section. Since these areas of design are known to be stress-raisers, knowledge of their effect on the strength of parts is increasingly important to designers. The largest segment of present knowledge on this subject deals with the effect of stress-raisers on the lives of laboratory-type specimens and has only limited design application. The fundamental laws governing fatigue cracking have therefore great practical as well as theoretical importance. Attempts to determine basic mechanisms have generally used metallographic sectioning to detect cracks. However, very shallow or very short cracks may not be detected by this method. Furthermore, it is impossible in this way to determine the extent of the cracks across the surface.

It was decided therefore to observe at high magnifications the surface initiation and propagation of fatigue cracks in the vicinity of notches. Quantitative measurements were made whenever possible in an attempt to discover the operative mechanisms. In this sense, the present investigation complements a previous one on unnotched specimens (**1**).[2]

## Material and Testing Method

All of the experimental observations were made on the same 6061-T6 aluminum alloy previously used to observe the propagation of fatigue cracks in unnotched specimens (**1**). This material had been obtained as commercial $\frac{3}{4}$-in. diameter rolled-and-drawn rod having a

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Assistant Chief, Metallography Division and Research Metallurgist, respectively, Alcoa Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 652.

TABLE I.—SPECIMEN DIMENSIONS.

| Notch, Radius, in. | Diameter, in. | | Flank Angle, deg | $K_t$ |
|---|---|---|---|---|
| | Max | Min | | |
| 9.875[a] | ... | 0.300 | ... | 1.00 |
| 0.500 | 0.480 | 0.330 | ... | 1.13 |
| 0.106 | 0.480 | 0.330 | 60 | 1.40 |
| 0.062 | 0.480 | 0.330 | 60 | 1.60 |
| 0.031 | 0.480 | 0.330 | 60 | 1.95 |
| 0.005 | 0.480 | 0.330 | 60 | 4.17 |
| 0.0002 | 0.480 | 0.330 | 60 | 18.9 |

[a] Smooth specimens reported in reference (**1**).

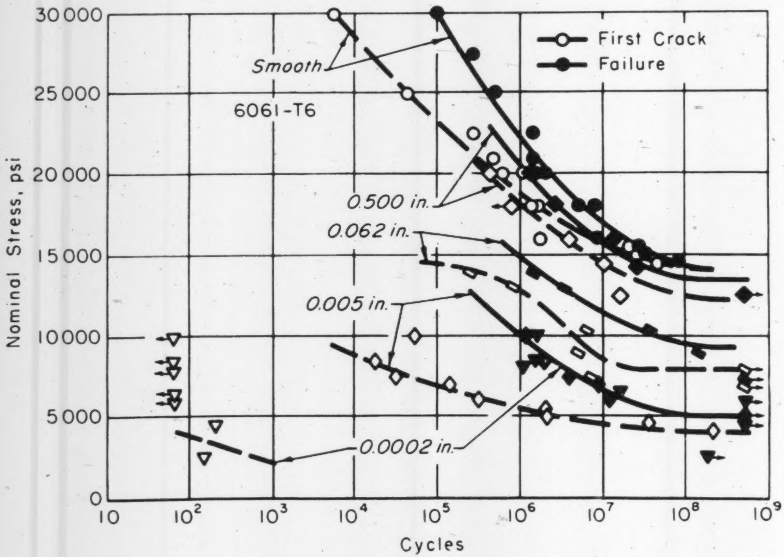

FIG. 1.—Relations between the First-Crack and Failure Curves of 6061-T6 Smooth and Notched Specimens. Notch radii are given in inches. Stress is based on minimum diameter making no allowance for stress concentration by notch. Data for 0.106 and 0.031-in. radius notches not plotted.

tensile strength of 44,700 psi, a yield strength (0.2 per cent offset) of 37,700 psi, and an elongation of 19.5 per cent in 2 in. Specimens were prepared with a series of circumferential notches ranging in severity from very mild to extremely severe. The effect of most of these notches on the fatigue life of other alloys had been established previously (**2**). Dimensions of the various notched specimens, as well as the smooth specimens used in Reference (**1**), are indicated in Table I.

In this investigation, as in the work described in Reference (**1**), each specimen was given a light chemical treatment before testing to obtain a metallographically polished surface. The fatigue testing, which was done in R. R. Moore rotating-beam machines, was interrupted periodically in order to obtain plastic replicas of the specimen surface. These replicas reproduced surface details faithfully and could be examined under the microscope at high magnifications. By comparing the series of replicas for any test, it was possible to observe the formation of all fatigue cracks at the notch root and to follow their surface propagation around the specimen.

The material used for making the replicas was cellulose acetate ribbon. By using narrow strips of ribbon softened in solvent, it was possible to obtain excellent replicas from notches with radii of 0.031 in. or greater. With sharper notches, a special technique was needed which took advantage of the thermoplastic properties of the ribbon. Replica blanks with a shape that matched the notch contour were molded from the plastic under heat and pressure. When these blanks were softened with solvent and pressed into the base of the notch, a good replica of the notch structure was achieved.

## TEST RESULTS

### *Crack Initiation:*

The first obvious effect of a notch was in the number of cycles at which cracks appeared. When the number of cycles at which a microscopic size crack first appeared in each specimen was noted on the *S-N* plot, the points for all tests of a particular notch severity were found to lie along a curve defining the first inception of cracking at various stress levels. The first-crack curves for the various specimen types are plotted in Fig. 1 along with the conventional *S-N* curves denoting the number of cycles at which final failure occurred. The stresses in this plot are nominal stresses based on the minimum diameter, making no allowance for stress concentration by the notch. For clarity, the data for the 0.106 and 0.031-in. radius notches are omitted from Fig. 1 but are included in later figures.

It will be noted from Fig. 1 that, at a given nominal stress, cracks were initiated earlier as the notches became sharper. In addition, the spread between the first-crack and failure curves, that is, the proportion of the total life consumed in the growth of the initial crack, increased with increasing notch severity. This observation agrees with one made by Bennett and Weinberg (3) in specimens containing fillets as stress raisers.

Two differences are apparent between the first-crack curves of the smooth and notched specimens. While the first-crack curve of the smooth specimens is smoothly convergent on the failure curve at the endurance limit, this is not true of the notched specimens. First, there was a tendency for the first-crack curve of some notched specimens to take a downward trend between one and ten million cycles before leveling off at the minimum stress necessary for cracking. Second, cracking occurred at stresses considerably below the notch endurance limit. Cracks were observed at lower nominal stresses and at a smaller fraction of the notch endurance limit as the notches became more severe.

The cracking data for the sharpest notch are incomplete. With this severity of notch, the dead weight load of the housings of the testing machine proved sufficient to cause cracks after only a few hundred cycles. This notch was so

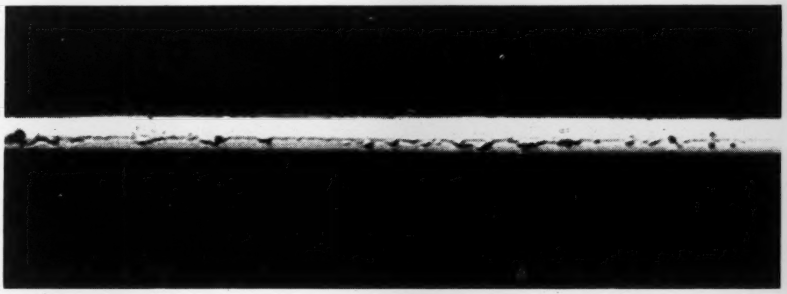

FIG. 2.—Evidence of Cracking at Base of 0.0002-in. Radius Notch after First Application of 8500 psi (× 750).

severe, in fact, that although no cracks were present before testing, there were definite indications of cracking at the very first application of any load sufficient to cause fatigue failure (Fig. 2).

The effective stress at the base of the notch is greater, of course, than the nominal stress of Fig. 1. When the data of this figure are replotted on the basis of the theoretical stresses calculated by the method of Neuber (**4**), Fig. 3 results. The effect of this operation is to concentrate all of the data marking inception of cracking into one area. While separate curves may be drawn for each notch severity, the differences between the various curves of the set would probably not be significant nor would there be a trend for displacement of the first crack curves with increasing severity, with the possible exception of the 0.005-in. radius notch. The bulk of the first crack data for notch specimens, however, lies somewhat to the right of the first-crack curve of smooth specimens.

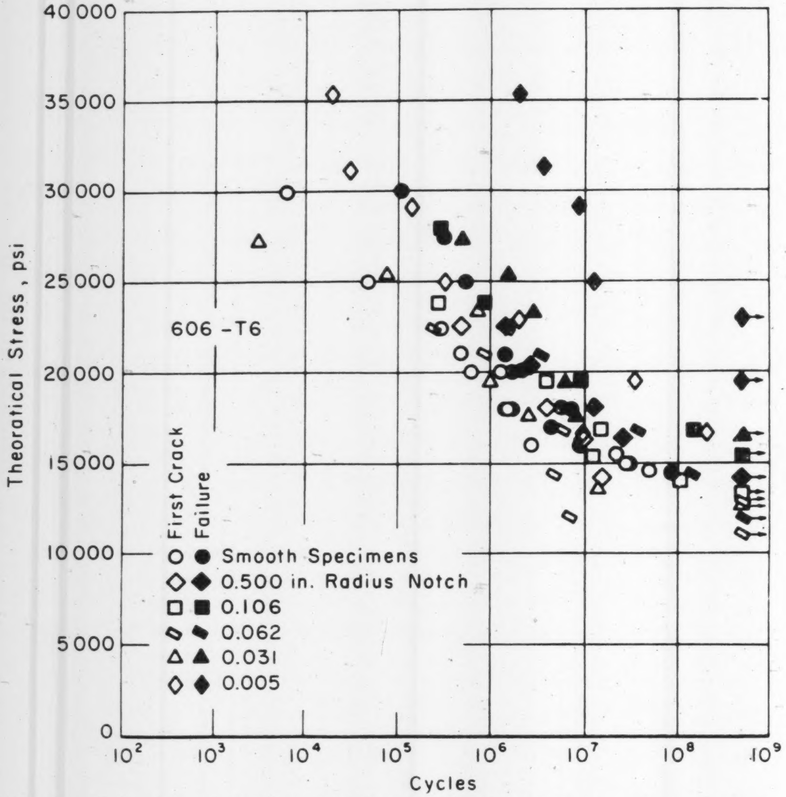

FIG. 3.—Data of Fig. 1 Replotted on Basis of Theoretical Stress at Base of Notch.

Plotting on the basis of the theoretical stress at the root of the notch produces coincidence of the first crack data but not of the failure data. The theoretical stress is a good criterion of failure for only those notches with a $K_t$ less than about 1.6 and becomes increasingly poorer with sharper notches. For the sharpest notch, all points lie completely off the range of values covered by Fig. 3. However, the points defining the inception of cracking in this notch are reasonable extrapolations of the other first-crack curves, while the failure curve of this type specimen is far removed from the other failure curves when plotted to theoretical stresses.

The occurrence of cracking at stresses below the notch endurance limits of the several specimen types is also explicable from Fig. 3. In general, when the theoretical stress at the notch root exceeded the stress at which cracking occurred in smooth specimens (the endurance limit of unnotched specimens) cracks appeared in the notch. When the theoretical stress was lower, cracking, with a few exceptions, did not occur. In the exceptional cases, cracking was associated with additional stress raisers such as small pits at the notch root, and the crack, once formed, did not grow to appreciable size.

*Crack Propagation:*

The presence of notches in these specimens had an effect on the rate of crack propagation as well as on crack initiation. The general mechanisms of propagation, however, were the same as in the case of smooth specimens (**1**).

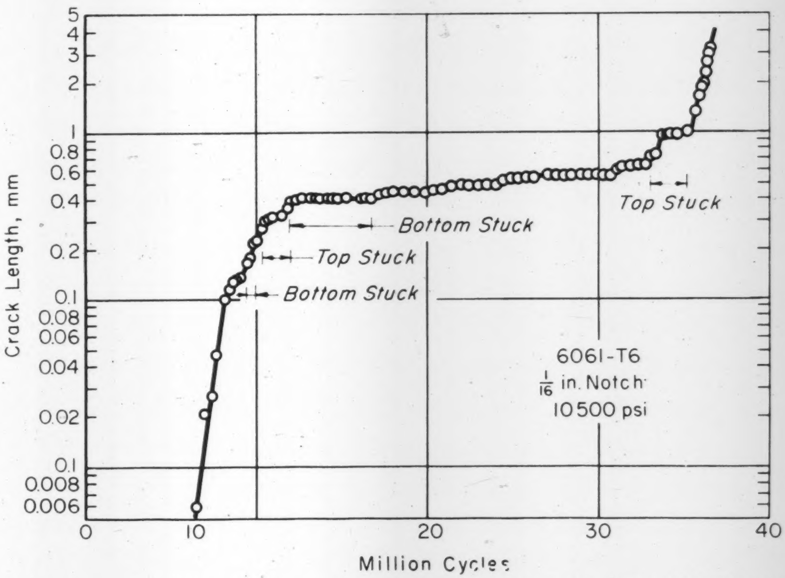

FIG. 4.—Typical Crack Growth Curve with Notations of Major Periods During Which One End of Crack Did Not Grow. The long horizontal portion of curve is the hesitation period.

A crack typically spread along the base of the notch, as illustrated in Fig. 4. The initial crack grew rapidly, accelerating as it grew larger until it reached a critical size between 0.1 and 1.0 mm (commonly about 0.3 mm) at which time it often stopped growing for an appreciable period. If the test stress was in excess of the notch endurance limit, it later resumed growing at the original rate. If the test stress was below this endurance limit, individual cracks did not pass out of the critical range.

The instantaneous lengths, *l*, of a crack while it was growing when plotted on semilogarithmic scales with the number of cycles, *N*, as in Fig. 4, fell along a straight line and consequently obeyed the relation

$$\log l = aN + b$$

where *a* and *b* are constants. This meant that a constant number of cycles was needed for a crack to increase in length by a given multiple. It is convenient to express the slope of the inclined portions of curves such as Fig. 4 in terms of the "doubling time," the number of cycles required for a crack to double in length.

Plotted in Fig. 5 in terms of the doubling times are the rates of crack propagation in the various notched specimens along with the rates previously determined from smooth specimens. It will be seen that specimens with the same theoretical stress at the notch root have the same doubling time, approximately that of the unnotched specimens, whenever the test stress exceeded the notch endurance limit. In cases in which cracks propagated to critical size below the endurance limit, propagation was much slower than typical of the theoretical stress.

Propagation did not continue indefinitely at the rates plotted in Fig. 5 in all cases. Commonly, when cracks in relatively sharp notches reached the size range at which cracks in milder notches hesitated in their growth, they assumed a slower rate of growth closer to that typical of smooth specimens at that nominal stress.

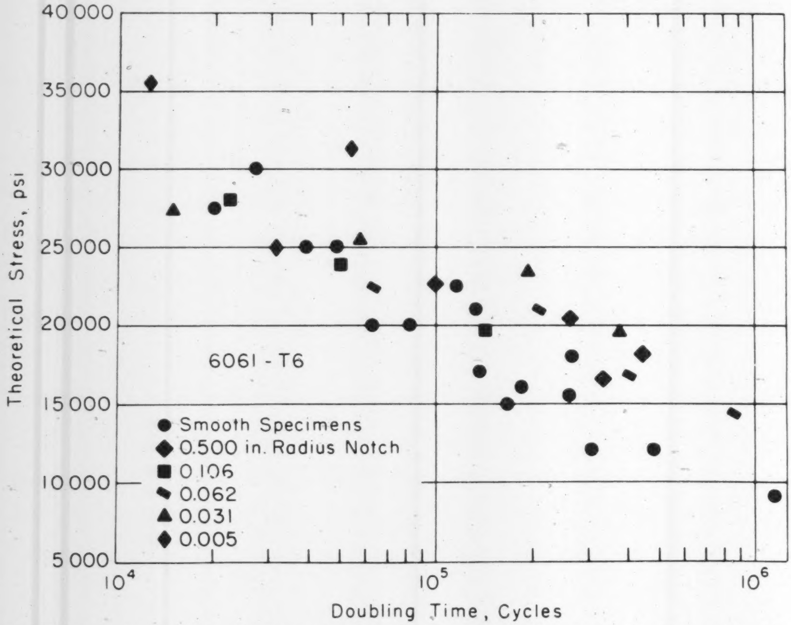

FIG. 5.—Rate of Crack Growth Given in Terms of Doubling Time, the Number of Cycles Needed for a Crack to Double in Length.

### *Crack Hesitation:*

Cracks growing in smooth specimens characteristically hesitate in their growth, sometimes for prolonged periods before propagating to failure (1). However, the period of hesitation at high stresses is of such short duration as to be relatively undetectable. Hesitation periods resembling those of smooth specimens were observed in the milder notches in this investigation. The long horizontal portion of the growth curve of Fig. 4 illustrates a typical hesitation period.

The number of cycles a crack remained immobilized was a function of the test stress, being larger at the lower stresses. Figure 6 gives some typical data. It will be seen that cracks in notched specimens tested at the same theoretical stress have similar hesitation periods that are somewhat longer than those in smooth specimens. The theoretical stress in the sharper notch specimens was generally too high for crack hesitation to be detected.

*Number of Cracks:*

A final effect of the presence of notches was in the number of cracks found in individual specimens. As the notches were made sharper at a given nominal stress, more cracks appeared before failure. This increase in the number of cracks was at least qualitatively a function of the theoretical stress.

One consequence of the profusion of cracks within the sharper notches was the decreasing number of cycles at which the complete specimen circumference became cracked. There is ample evidence in the literature that circumferential cracking occurs early in sharply notched specimens (5,6). This investigation showed that this is the result of the joining of cracks from many nuclei. The tendency was, moreover, reflected in the appearance of the fracture surfaces after final failure. The portion of the fracture having markings typical of static failure was more centrally located near the specimen axis as the notches became sharper and the nominal stresses became higher, two conditions that raised the theoretical stress and provided more embryonic fatigue cracks around the notch circumference.

A paradoxical situation existed in the

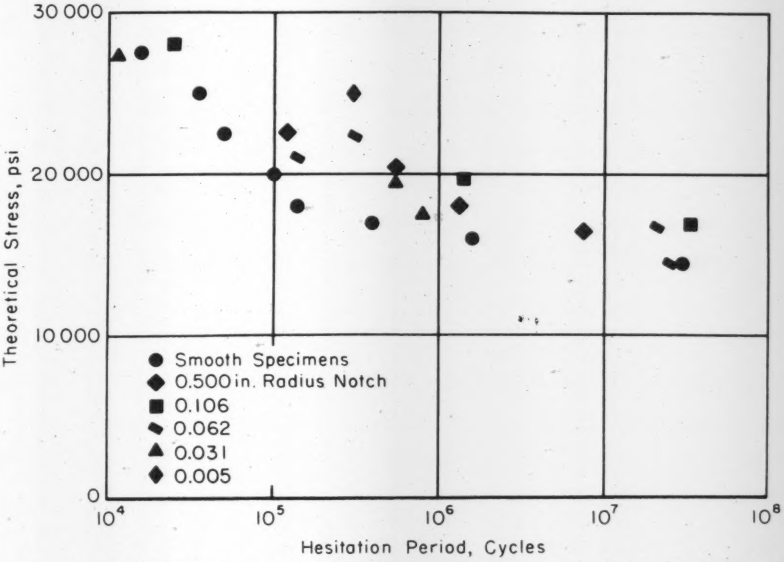

FIG. 6.—Typical Hesitation Periods During Which Propagating Cracks Did Not Grow.

case of specimens stressed below the endurance limit. It is customarily assumed that as the test stress is reduced the number of fatigue cracks is likewise reduced. This was true in this investigation as long as observation was confined to specimens that eventually failed. When the test stress was insufficient to cause failure, however, the very large number of cycles imposed on the specimen permitted cracking in some areas that originally were strong enough to withstand a limited number of cycles. There existed, therefore, stress ranges where a larger number of cracks were eventually produced than at somewhat higher stresses. None of the cracks individually grew to large size, but it was possible with comparatively sharp notches to have the complete circumference effectively cracked with many-branched, feathery cracks although failure never occurred (Fig. 7).

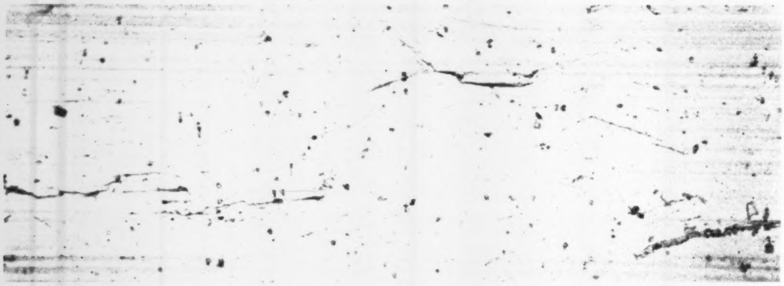

FIG. 7.—Specimen Stressed Below Notch Endurance Limit (× 100). Many short cracks were formed because of large number of cycles imposed. Stress 8500 psi, 0.031-in. radius notch, 5 × $10^8$ cycles without failure.

It is important to recognize that the "non-propagating" cracks formed below the endurance limit remain extremely shallow. They have no detrimental effects on static properties as demonstrated in tension tests on some of the present specimens (Table II). All specimens previously tested in fatigue contained cracks around the entire circumference.

## DISCUSSION

As shown quantitatively by the times for crack initiation, the rates of crack propagation, and the times of crack hesitation and qualitatively by the number of cracks, the effective stress at the base of the notches in this experiment was that calculated from the theoretical stress concentration factor, $K_t$, or was just slightly less than the theoretical stress. Except for the milder notches, the operative stress was greater than that computed from a strength reduction factor, $K_f$, which is based on failure rather than cracking data. This confirms a similar finding by Bennett and Weinberg (3) in specimens containing fillets and lends experimental justification to

TABLE II.—STATIC TESTS OF CRACKED FATIGUE SPECIMENS.
(0.0002 in. radius notch)

| Previous Fatigue History | | Apparent Tensile Strength, psi[a] |
|---|---|---|
| Nominal Stress, psi | Cycles | |
| 6000 | 532 313 000 | 64 800 |
| 4700 | 513 000 000 | 65 300 |
| 2600 | 177 262 000 | 64 900 |
| ... | ... | ... |
| Not subjected to fatigue | | 65 300 |

[a] Based on diameter at base of notch.

Peterson's suggestion (7) that $K_t$ be used in the design of large structures.

These observations of the importance of the theoretical stress in the early stages of crack initiation and surface propagation suggest that explanations of differences in lives of different specimen types tested at the same theoretical stress should be sought in the mechanics of crack deepening. There exists, for example, evidence that shallow cracks form quickly, but that these do not deepen until shortly before failure (5,6). The comparative difficulty with which deepening cracks leave the "field" of the various notches may explain why in this experiment specimens containing 0.0002-in. radius notches survived as long as those containing 0.005-in. radius notches, even though cracks formed earlier. In other investigations, specimens with the sharpest notches have, in fact, proved to be longer lived than those with slightly milder notches(5,8).

The fact that cracks form in notched specimens at stresses below the endurance limit has, of course, been known for some time. Lessells and Jacques (9), for example, found them at stresses only 45 per cent of the endurance limit. Previous investigations, however, have been restricted to examination of metallographic sections. The surface examinations of the present experiment have permitted the detection of cracks too shallow to be seen in polished sections as well as those so short as to lie out of the sectioning plane. This may explain why Frost and Phillips (5) found no "non-propagating cracks" in notches with $K_t$ less than about 9, while the present investigation disclosed them in even the mildest notch.

The present finding that cracking at the notch root occurs when the theoretical stress exceeds the endurance limit of smooth specimens approximates Phillips' finding (10) that "non-propagating cracks" deepen through all the material in which the theoretical stress exceeds the endurance limit. The significance of the endurance limit itself is made more apparent by the observation that large numbers of cracks form in specimens in which the accumulation of many cycles is not prevented by early fracture. Apparently, the virgin material contains many areas of differing strength, each requiring a definite number of cycles to initiate cracking at a given test stress. Even the weakest area, however, can resist a stress equal to the endurance limit for an infinite number of cycles.

It might seem surprising that a material as ductile as 6061-T6 showed indications of cracking at the first application of comparatively low loads when the notch was very sharp, even granting that the theoretical stress far exceeded the ultimate strength of the alloy. Fenner, Owens, and Phillips (11), however, have previously suspected the presence of these very early cracks in a steel using a magnetic detection method. Clearly, more work is needed in the field before generalizations can be made. The problem is, however, of theoretical rather than practical interest, for the extremely shallow cracks formed have no effect on either the static or fatigue properties of the material.

## CONCLUSION

This investigation has shown that the theoretical stress obtained at notches is a good, though slightly conservative, measure of the stress controlling fatigue crack initiation and the early stages of crack propagation. This would indicate that structures should be designed on the basis of theoretical stress if even the most minute fatigue cracks could not be tolerated.

The problem remaining is that of determining whether the findings of the present investigation hold true for other alloys, especially those known to be notch sensitive. More important from a practical standpoint, however, is a determination of the modes and rates of crack propagation away from the vicinity of stress-raisers and of the effect of the presence of very small fatigue cracks on mechanical properties. Only with this information will it be possible to approach the design problem with absolute confidence.

*Acknowledgment:*

The authors express their appreciation to F. Keller under whose direction this investigation was conducted and to L. L. McMaster and B. A. Walker of the Mechanical Testing Division of the Alcoa Research Laboratories who conducted the fatigue tests.

## REFERENCES

(1) M. S. Hunter and W. G. Fricke, Jr., "Fatigue Crack Propagation in Aluminum Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 1038 (1956).

(2) R. L. Templin, "Fatigue of Aluminum," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 641 (1954).

(3) J. A. Bennett and J. G. Weinberg, "Fatigue Notch Sensitivity of Some Aluminum Alloys," *Journal of Research*, Nat. Bureau Standards, Vol. 52, p. 235 (1954).

(4) H. Neuber, "Theory of Notch Stresses, Principles for Exact Stress Calculations," Translation by David Taylor Model Basin (1946).

(5) N. E. Frost and C. E. Phillips, "Studies in the Formation and Propagation of Cracks in Fatigue Specimens," International Conference on Fatigue of Metals, Inst. Mechanical Engrs. (1956).

(6) W. S. Hyler, E. D. Abraham and H. J. Grover, "Fatigue Crack Propagation in Severely Notched Bars," *Technical Note No. 3685*, U. S. Nat. Advisory Comm. for Aeronautics (1956).

(7) R. E. Peterson, "Relation Between Stress Analysis and Fatigue of Metals," *Proceedings*, Soc. Experimental Stress Analysis, Vol. 11, No. 2, p. 199 (1954).

(8) J. Y. Mann, "The Effect of Stress Concentrations on the Fatigue Resistance of 24S-T Aluminum Alloy," *Report SM217*, Australian Aero. Res. Lab., Nov., 1953.

(9) J. M. Lessells and H. E. Jacques, "Effect of Fatigue on the Transition Temperature of Steel," *Welding Journal*, Research Supplement, Vol. 29, p. 74s (1950).

(10) C. E. Phillips, "Fatigue Cracks as Stress Raisers, and Their Response in Cyclic Loading," Fatigue in Aircraft Structures, Academic Press, p. 104 (1956).

(11) A. J. Fenner, N. B. Owen, and C. E. Phillips, "The Fatigue Crack as a Stress Raiser," *Engineering*, Vol. 171, p. 637 (1951).

# DISCUSSION

MR. JOHN A. BENNETT.[1]—There is one point in regard to nonpropagating cracks that I believe should be mentioned here. These considerations apply to rotating-beam or other types of reversed stress tests with sharply notched specimens.

The left side of the accompanying Fig. 8 represents the cross-section of a notched specimen with a fatigue crack that has grown to a depth several times the radius of the notch. During the tensile half of the cycle the crack is opened and the stress at the end of the crack is very high. However, when the stress is compressive the two sides of the crack are pressed together and the stress distribution is much the same as it was before the crack developed. Because the crack has progressed out of the region of high stress concentration due to the notch, the compressive stress at the end of the crack is only slightly higher than the nominal value.

The right side of the figure illustrates this schematically, assuming that the stress concentration due to the crack is the same as that due to the notch (which would be approximately true with the sharp notches used by the authors). The solid line shows the stress cycle at the bottom of the notch before the crack develops. As the crack progresses away from the region where the notch causes high stress concentration, there is no change in the stress acting at the crack front during the tension part of the cycle but it is sharply reduced during the compressive part, as indicated by the dashed line. The result is that the stress amplitude which is effective in extending the crack is only a little more than half that which caused its initiation. This may be an important consideration in explaining the non-propagating cracks in this type of test.

With this in mind, it would be interesting to know what happens in unidirectional tests where the stress does not go into compression. I wonder if the authors are planning to do work of this kind.

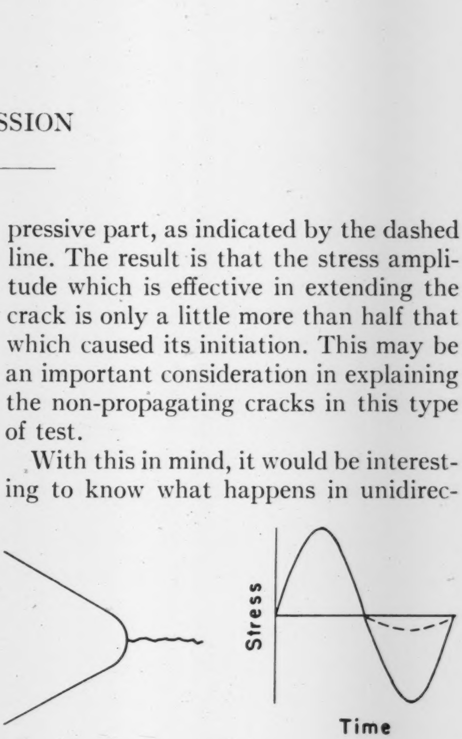

FIG. 8.

MR. DAVID E. MARTIN.[2]—I would like to comment, in connection with Mr. Bennett's suggestion, that the hesitation period observed by the authors was due to the crack closing on the compression half of the load cycle. Mr. Bennett remarked that under unidirectional tensile cycling there should be no hesitation period, as the crack would always be open and behave as a stress raiser. We have measured crack growth in sheet specimens under unidirectional loading at the University of Illinois and have not observed a hesitation period. These measurements were made on specimens 3 in. wide and 0.032 in. thick. The cracks were first observed as small as 0.004 in. long and were measured periodically until one cycle prior to fracture, at which time the cracks were as long as 1.5 in.

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[2] Special Problems Department, Research Staff, General Motors Corp., Detroit, Mich.

I would like to ask the authors if they computed the theoretical stress from the original notch geometry (or from the crack length) and if the length of the cracks seemed to influence the propagation rates.

MR. PAUL KUHN.[3]—I am somewhat surprised that no Templin notches were included, with a radius of 0.0002 in. If I remember correctly, the smallest radius shown was 0.005 in. If the Templin notch had been included, it may be that the use of the theoretical stresses would not look so well.

MR. H. C. BURNETT.[4]—I should like to take this opportunity to make a plea for more work along the lines of this paper. I feel that designers would be greatly assisted if fatigue data included not only the cycles to failure but also the cycles to when the first crack may be detected. This is particularly applicable in spring design, where the initial crack is generally parallel to the axis of the wire and greatly reduces the spring constant or load-carrying characteristics of the spring. In that instance the start of the first crack is important to designers. If we get information on when cracks start, it would be beneficial to report it.

MESSRS. M. S. HUNTER AND W. G. FRICKE, JR. (*authors' closure*).—We are indebted to Mr. Bennett and Mr. Martin for the discussion of crack hesitation in axially-loaded fatigue specimens. We have also become interested in the problem and anticipate making observations during this type of test. It is important, however, in discussing hesitating or nonpropagating cracks to differentiate between cracks propagating away from a stress raiser in the direction of decreasing external stress, such as those studied by Phillips[5] and cracks propagating along stress raisers in uniform external stress fields such as those reported in this paper. The relation between the two types is not clear.

The theoretical stresses reported in the paper are computed from the initial notch geometry, and no modifications were made to these values to account for the presence of a crack. The presence of a crack may not, in fact, profoundly influence the original stress distribution, considering that the rate of crack propagation just before failure is not very different than that of the initial crack.

The significance of data based upon theoretical stress in the very sharp 0.0002-in. radius Templin type notches is obscure. Theoretical stresses for this sharp a notch are approximations at best and exceed the ultimate strength of the material for even the lowest loads used. It is true that the first-crack data of this notch are of the right order of magnitude to be extrapolations of the data for the other notches when plotted to theoretical stress. This, however, necessitates extrapolation over large distances so that the procedure is open to question. Stresses were so severe with the Templin notch that it was experimentally impractical to measure rates of crack propagation or observe crack hesitation.

Mr. Burnett's plea for more cracking data is well founded. Designers and users of structures are very interested in learning when cracks are present in a part. There remains, however, the question of knowing how harmful a crack is, especially in the case when the crack is an extremely shallow one in the vicinity of a stress raiser and can be detected only by special metallographic techniques.

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[4] Metallurgist, National Bureau of Standards, Washington, D. C.

[5] C. E. Phillips, "Fatigue Cracks as Stress Raisers and Their Response to Cyclic Loading," Fatigue in Aircraft Structures, Academic Press (1956).

# AXIAL-LOAD FATIGUE PROPERTIES OF HIGH-STRENGTH STEELS

BY B. B. MUVDI,[1] G. SACHS,[2] AND E. P. KLIER[3]

## SYNOPSIS

This report presents the results of axial-load (completely reversed) fatigue tests on Tricent, Cru. UHS-260, and Super TM-2 steels heat treated to strength levels between approximately 250,000 and 300,000 psi. The fundamental effects of several variables on the fatigue properties of these steels are discussed and evaluated. These variables included the notch sharpness or the stress concentration, the strength level and the specimen orientation.

The results indicated that the fatigue strength was lowered as the stress-concentration factor was increased, a maximum lowering effect occurring for stress concentration factors between 1 and 3. This effect was observed to depend upon the strength level. The endurance limit for both smooth and notched specimens developed minimum values at a strength level between 240,000 and 260,000 psi and maximum values between 270,000 and 300,000 psi approximately. In general, the endurance limit was found to be lower for transverse than for longitudinal specimens. Furthermore, this effect was much more pronounced for smooth than for notched specimens and was observed to be severe at high strength levels and to decrease with decrease in this quantity.

The repeated occurrence of fatigue failures in steel parts heat treated to strength levels above 200,000 psi has resulted in the belief that steels heat treated to such high strength values possess fatigue properties inferior to those of steels in the range below 200,000 psi (**1**).[4] However, a number of recent investigations (**1**,**2**,**3**) have demonstrated, contrary to common concepts, that high-strength steels may exhibit endurance strength values considerably superior to those of steels heat treated to a strength of 200,000 psi or lower. The metallurgical considerations alone are, therefore, insufficient for the prediction of performance, and other factors, particularly those resulting from certain design requirements, cannot be ignored.

In a previous investigation performed at Syracuse University (**3**), rotating-beam fatigue tests were performed on a number of high-strength aircraft steels. The results indicated that while the endurance ratio of both smooth and notched specimens decreased, the endurance limit, in general, slightly increased with increase in the tensile strength above 200,000 psi.

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[2] Associate Director, Syracuse University Research Inst., Syracuse, N. Y.

[3] Professor of Metallurgy, Syracuse University, Syracuse, N. Y.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 666.

In this investigation the fundamental effects of a number of variables on the fatigue properties of high-strength steels were examined. Tension-compression ($R = -1$) fatigue tests were performed on specimens machined from 3 to $4\frac{1}{2}$ in. (round or square) sections. The strength range investigated varied between 250,000 and 300,000 psi approximately. One to three strength levels were investigated in each of Tricent, Cru. UHS-260 and Super TM-2 steels. In all instances specimens both longitudinal (parallel to the rolling direction, symbol $L$) and transverse (perpendicular to the rolling direction, symbol $T$) were tested. Unnotched specimens ($K_t = 1$) and specimens provided with notches leading to stress concentration factors, $K_t = 3$, 5, and 8 were examined.

TABLE I.—COMPOSITION AND SIZE OF THE VARIOUS STEELS EXAMINED.

| Alloy | Size and Shape | Per cent of Alloying Elements | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Carbon | Manganese | Phosphorus | Sulfur | Silicon | Nickel | Chromium | Molybdenum | Vanadium | Copper |
| Tricent (Inco)......... | 4¼-in. square | 0.39 | 0.74 | 0.014 | 0.014 | 1.54 | 1.83 | 0.83 | 0.38 | 0.07 | .. |
| Cru. UHS-260......... | 4-in. square | 0.35 | 1.20 | 0.023 | 0.017 | 1.62 | .. | 1.26 | 0.32 | 0.20 | .. |
| Super TM-2........... | 3-in. round | 0.41 | 0.72 | 0.012 | 0.014 | 0.61 | 2.08 | 1.15 | 0.44 | .. | 0.14 |

## EXPERIMENTAL PROCEDURE

### *Materials:*

The steels examined were hot-rolled sections from commercial electric-furnace heats of Tricent, Cru. UHS-260 and Super TM-2. The chemical analyses and the as-processed section sizes of these steels are presented in Table I.

### *Test Specimen:*

The fatigue and notch-fatigue specimens used in this investigation are presented in Fig. 1. They were rough machined to approximately 0.015 in. oversize on each surface and finished to proper dimensions after heat treatment. Smooth specimens were mechanically polished by turning the specimen against a fine polishing belt moving parallel to the specimen and tightly pressed along its contour. Notches, on the other hand, were finish machined by means of a properly ground carbide tool.

The specimens were heated to the recommended austenitizing temperature for each steel and maintained at this temperature for a period of 1 hr. They were then oil quenched, tempered for a period of 4 hr. and air cooled.

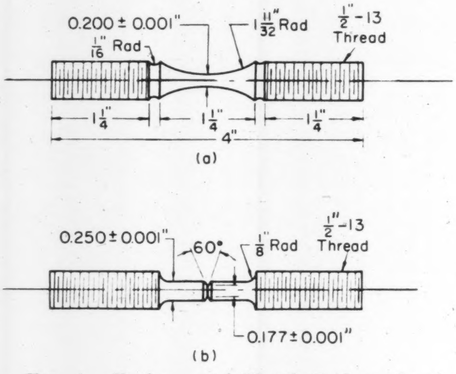

FIG. 1.—Fatigue and Notch-Fatigue Specimens Used in This Program.
(*a*) Smooth specimen. (*b*) Notched specimen.

### *Fatigue and Notch Fatigue Tests:*

All fatigue and notch fatigue tests performed in this investigation were of

TABLE II.—STRESS-CONCENTRATION FACTORS AND CORRESPONDING NOTCH RADII FOR NOTCH-FATIGUE SPECIMENS.

| Unnotched Diameter, D, in. | Notch Diameter, $d$, in. | $K_t$ | Notch Root Radius, $r$, in. |
|---|---|---|---|
| 0.250 | 0.177 | 3 | 0.009 |
| | | 5 | 0.003 |
| | | 8 | 0.001 |

5, and 8. The radii at the notch root corresponding to these stress-concentration factors were computed by Neuber's theory according to Peterson (4) and are given in Table II.

A 1000-lb Sonntag fatigue testing machine was used. By means of a special amplification device, Fig. 2, this machine was converted into an axial-load fatigue machine, capable of exerting a maximum load of ± 8000 lb. To insure concentricity of loading, the fixture shown in Fig. 3 was used. The assembly, consisting of the specimen and the aligning fixture, was aligned by bringing the two outer plates (marked $A$) into parallelism by adjusting one or more of the six adjusting screws. The whole assembly was then placed between the two parallel platforms of the amplification fixture, Fig. 2, and screwed to them, care being taken not to disturb the alignment of the specimens.

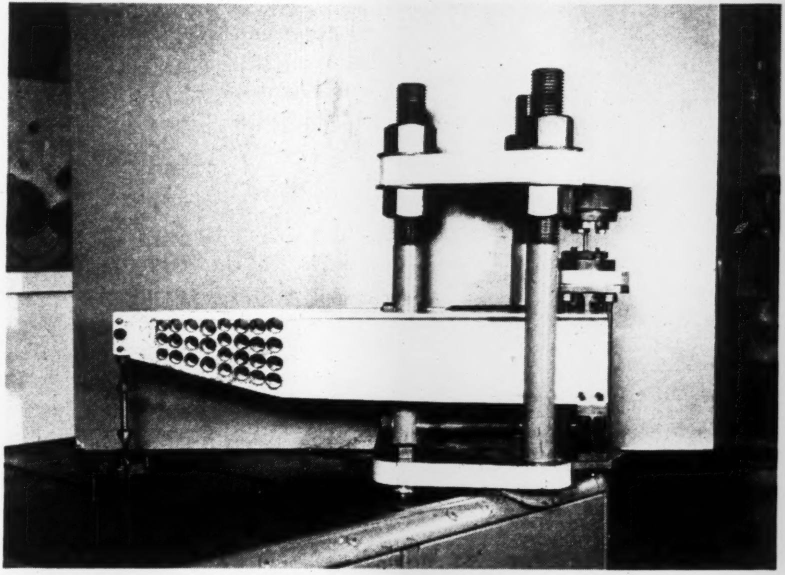

FIG. 2.—Amplification Fixture.

the tension-compression type with the stress ratio maintained constant at $R = -1$.

The smooth specimen used in this study is illustrated in Fig. 1($a$) and the notched specimen in Fig. 1($b$).[5] The notch on the latter specimen was of the 60-deg, 50-per cent reduction of section type. Three different stress concentration factors were employed, namely $K_t = 3$,

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[5] The 3-in. diameter Super TM-2 steel section was forged, by upsetting in the rolling direction, into 4-in. square slabs for the preparation of 4-in. long transverse specimens.

## EXPERIMENTAL RESULTS

### *Experimental S-N Curves for Smooth and Notched Specimens:*

The results of axial-load fatigue tests performed on longitudinal and transverse, both smooth and notched, specimens are presented in Fig. 4(*a*, *b*, *c*, *d*) and Fig. 5(*a*, *b*) for Tricent steel; in Fig. 5(*c*, *d*) and Fig. 6(*a*, *b*) for Cru. UHS-260 steel; and in Fig. 6(*c*, *d*) for Super TM-2 steel. The curves were accurately established for the range of $10^2$ to $10^6$ cycles. No experimental values could be obtained for less than 100 cycles as the equipment reached full speed and load only after about 50 cycles, and the counter recorded only in multiples of 100 cycles.

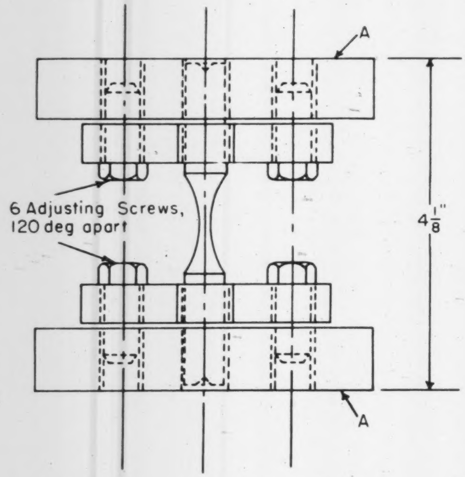


FIG. 3.—Assembly of Aligning Fixture and Fatigue Specimen.

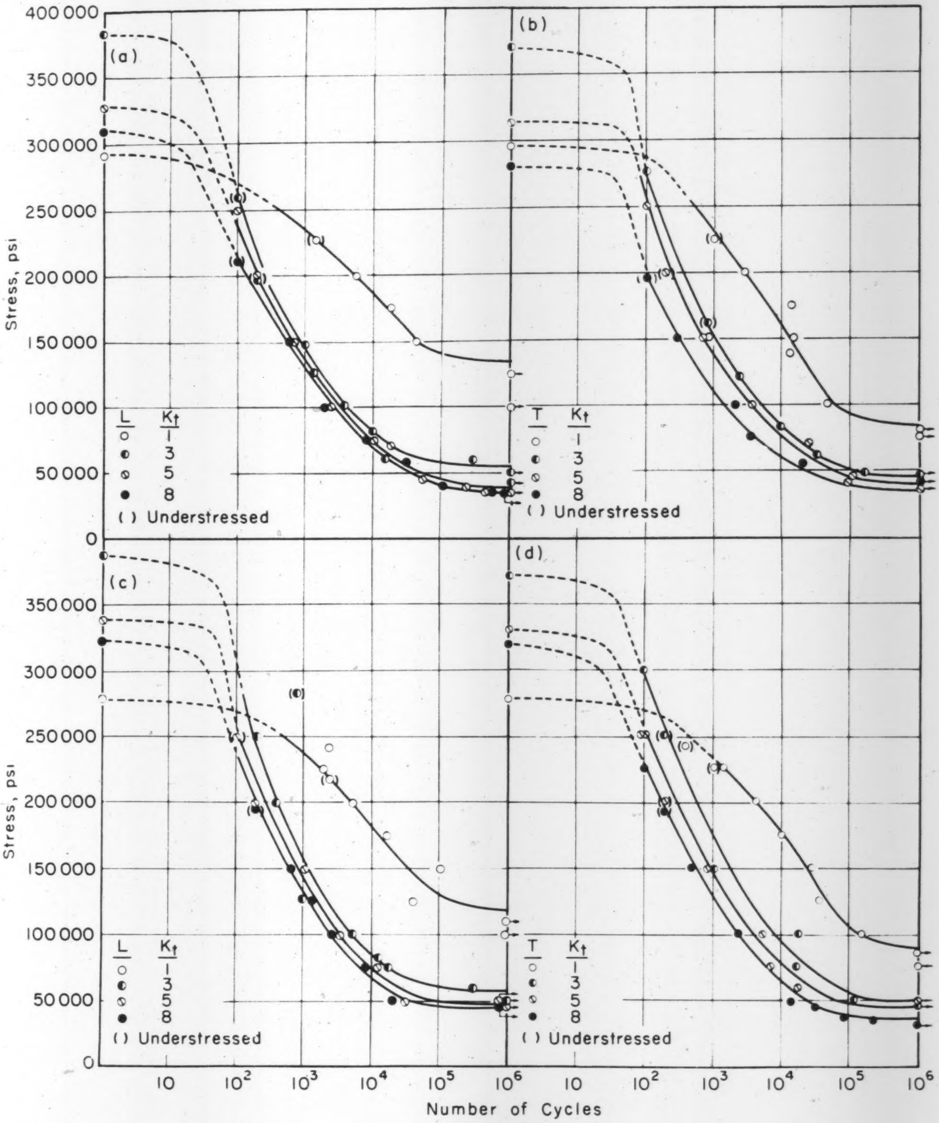
However, previous investigations on both smooth and notched specimens, (5, 6, 7) indicate that the direct-stress fatigue strength of various metals, including high-strength steels, differs only slightly from the tensile strength of the material, if the number of cycles to failure is less than about 100. These fatigue data also agree well with the concept that the tensile strength represents the origin of the *S-N* curve, in spite of the quite different rate of loading in the static and dynamic tests, respectively.

The curves in Figs. 4 to 7 are, therefore, extended by dotted lines to lower cycles, indicating their expected position in this range which could not be experimentally covered. The only exceptions in this respect are for a few transverse specimens of Cru. UHS-260. This discrepancy is explained by the fact that a different heat was used for the fatigue test than for the tension and notch-tension tests.

It is further observed that the direct-stress *S-N* curves for smooth and notched specimens, respectively, usually intersect or come very close to doing so. This also applies to the highest strength levels investigated (Fig. 4(*a*)) and to transverse specimens (Fig. 4(*b*)). In contrast, the previously obtained bending-fatigue *S-N* curves of the highest strength levels extrapolated to static (bending) strength values which were considerably higher for smooth than for notched specimens (3, Fig. 14). This was ascribed to the fact that the nominal bending strength of a smooth specimen having a high ductility should be much in excess of its tensile strength, while such "bend-strengthening" may be expected to be rather small in the case of notched specimens having low ductility.

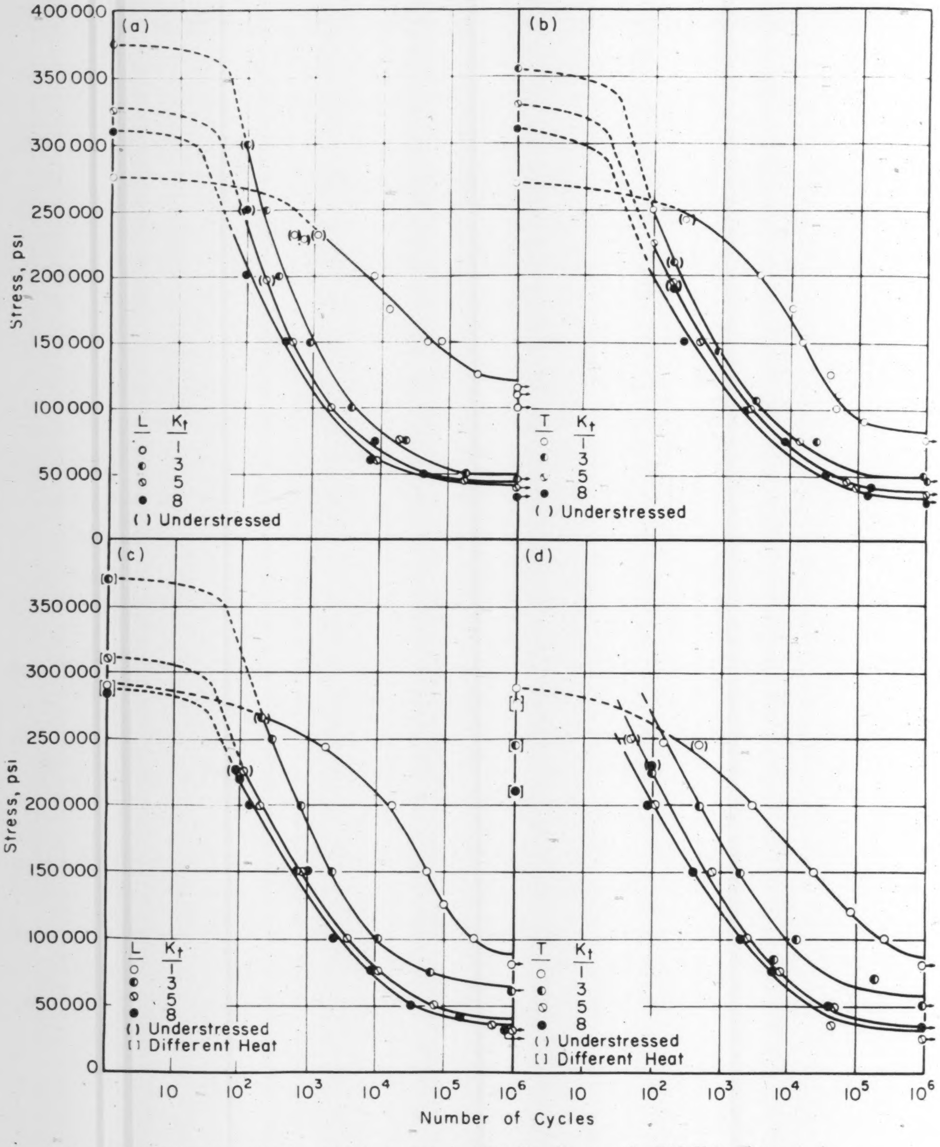
The axial-load fatigue strength of notched specimens is observed to fall below that of smooth specimens in the range above 100 cycles approximately. In general, the difference between notch- and smooth-fatigue strengths increases to a maximum value at about $10^4$ cycles and decreases with further increase in the number of cycles.

Below about 100 cycles, however, the notch-fatigue strength can be above or below the smooth-fatigue strength, ap-



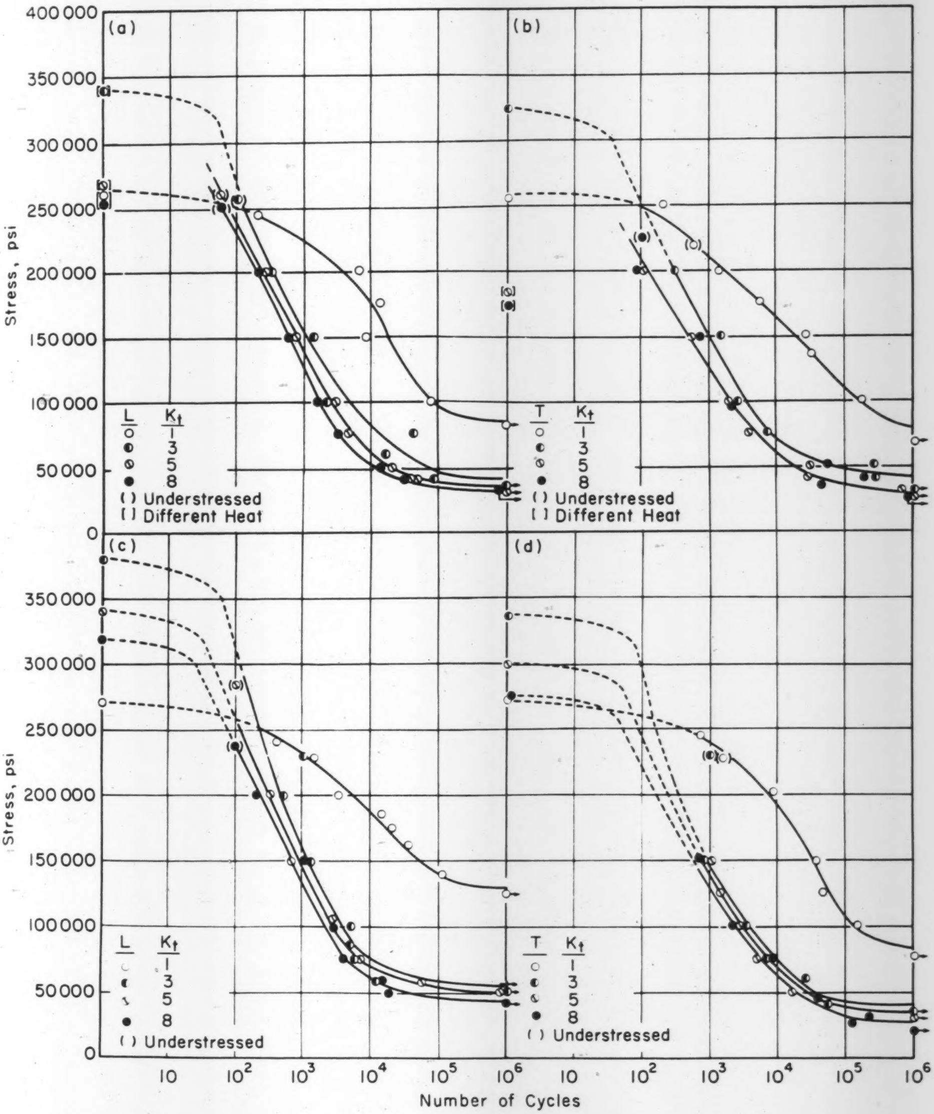
(*a*) Longitudinal specimens, 1600 F, oil quench, 400 F temper.
(*b*) Transverse specimens. 1600 F, oil quench, 400 F temper.
(*c*) Longitudinal specimens. 1600 F, oil quench, 550 F temper.
(*d*) Transverse specimens. 1600 F, oil quench, 550 F temper.

FIG. 4.—*S-N* Curves for Smooth and Notched Specimens of Tricent Steel.



(*a*) Longitudinal specimens of tricent steel, 1600 F, oil quench, 700 F temper.
(*b*) Transverse specimens of tricent steel, 1600 F, oil quench, 700 F temper.
(*c*) Longitudinal specimens of crucible UHS 260 steel, 1700 F, oil quench, 550 F temper.
(*d*) Transverse specimens of crucible UHS 260 steel, 1700 F, oil quench, 550 F temper.

FIG. 5.—*S-N* Curves for Smooth and Notched Specimens of Steel.



(*a*) Longitudinal specimens of crucible UHS 260 steel. 1700 F, oil quench, 800 F temper.
(*b*) Transverse specimens of crucible UHS 260 steel. 1700 F, oil quench, 800 F temper.
(*c*) Longitudinal specimens of super TM-2 steel. 1600 F, oil quench, 500 F temper.
(*d*) Transverse specimens of super TM-2 steel. 1600 F, oil quench, 500 F temper.

FIG. 6.—*S-N* Curves for Smooth and Notched Specimens of Steel.

proaching the static notch-tension strength at 1 (or $\frac{1}{4}$) cycle. This static notch-tension strength can be greater or less than the tensile strength depending upon the ductility and notch sensitivity of the steel (3).

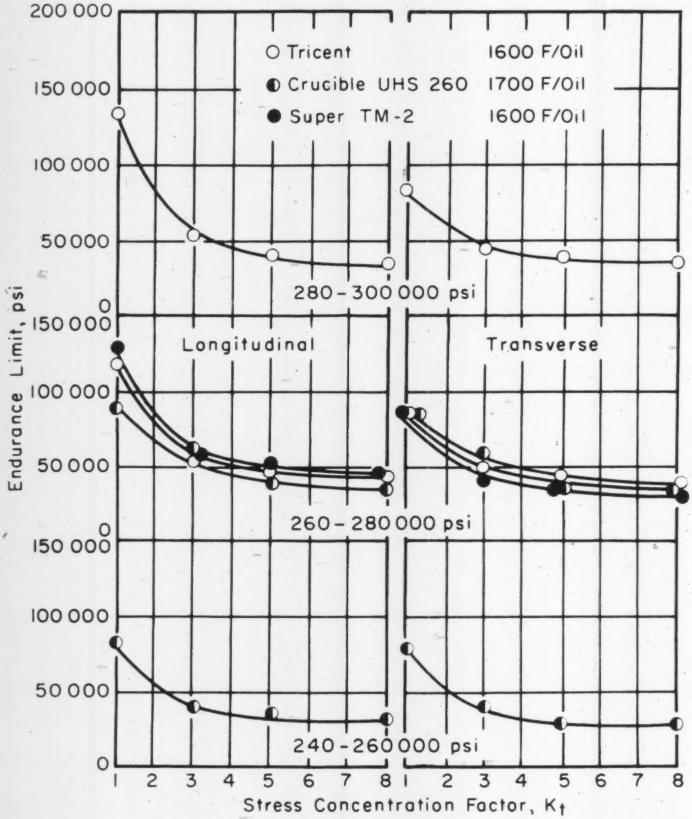


FIG. 7.—Dependence of Endurance Limit of High-Strength Steels on the Stress Concentration.

### *Effect of Stress Concentration on Endurance Limit of High-Strength Steels:*

As previously mentioned, the fatigue program included tests on smooth specimens and on specimens provided with notches leading to stress concentration factors of 3, 5, and 8. The results of these tests are presented in Fig. 7 as the endurance limit *versus* the stress concentration for both longitudinal and transverse specimens. These results clearly reveal that the effect of stress concentration on the endurance limit is most pronounced when the stress concentration is between 1 and 3 and at high rather than at low strength levels. Beyond a value of 3, the effect of stress concentration is minimized and the curves appear to approach a limiting value of endurance limit asymptotically. This behavior is equally true of the three strength ranges indicated and of both longitudinal and transverse specimens. However, it appears that the effect of the critical range of stress concentration ($K_t$ = 1 to 3) on the endurance limit is more pronounced in the longitudinal than in the transverse direction.

In the previous work on similar steels, heat treated to very high strength levels (3), rotating-beam bending-fatigue tests were performed for the same stress concentrations as used here. These regularly yielded minimum values of endurance limit for a stress concentration of $K_t = 3$, as shown for one example in Fig. 8. However, it was already pointed out at that time that these specimens ($K_t = 3$) were finished by grinding while all other notched specimens ($K_t = 5$ and 8) were finish-machined with a carbide tool. In contrast, maximum notch sensitivity at an intermediate stress concentration ($K_t = 3$) was absent in the direct-stress fatigue tests reported here. These specimens were all finish-machined. While the difference in test procedure—bending *versus* direct stress—renders a definite statement impossible, it now becomes very probable that grinding, in contrast to finish machining, of a notch decreases the endurance limit of super-high-strength steels by as much as 20,000 psi or 30 per cent.

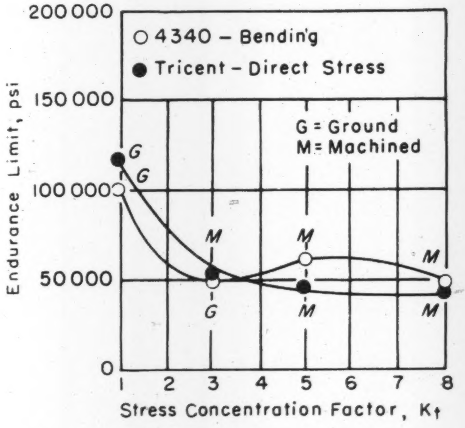


FIG. 8.—Effects of Grinding and Machining of Notches on the Endurance Limit of High-Strength Steels Heat Treated to 260,000 to 280,000 psi.

The effect of stress concentration is further illustrated in Fig. 9 where the ratio of endurance limit of smooth to that of notched specimens is plotted against the stress concentration factor using the tempering temperature as parameter. Only longitudinal properties are considered in this graph since transverse properties are, in general, subject to a greater degree of scattering. Previous tests on high-strength steels related generally to milder notches (1, 2), which revealed that the ratio of the endurance limits of smooth to notched specimens was generally equal to the stress concentration for mild notches, but became appreciably less than this as the stress concentration was increased. The results obtained in this program were, in general, observed to agree well with previous evidence.

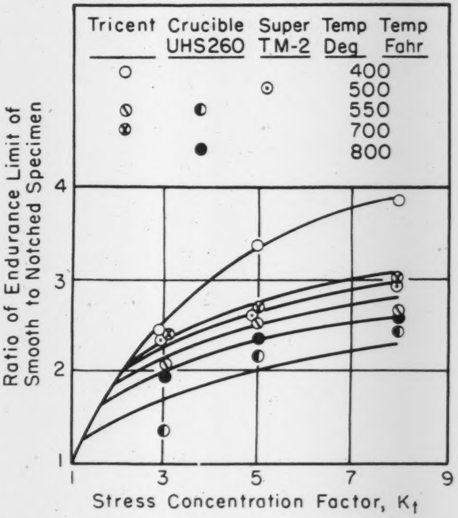


FIG. 9.—Effect of Stress Concentration on the Notch Sensitivity of High-Strength Steels.

Figure 9 indicates that the ratio of the endurance limits of smooth to notched specimens increases rapidly as the stress concentration factor is increased from 1 to 3. As the stress concentration is increased above $K_t = 3$, this ratio continues to increase at slower rates which vary with the steel composition and the tempering temperature.

steel tempered at 500 F. Cru. UHS-260 possesses the least sensitivity to notching, particularly when tempered at 550 F.

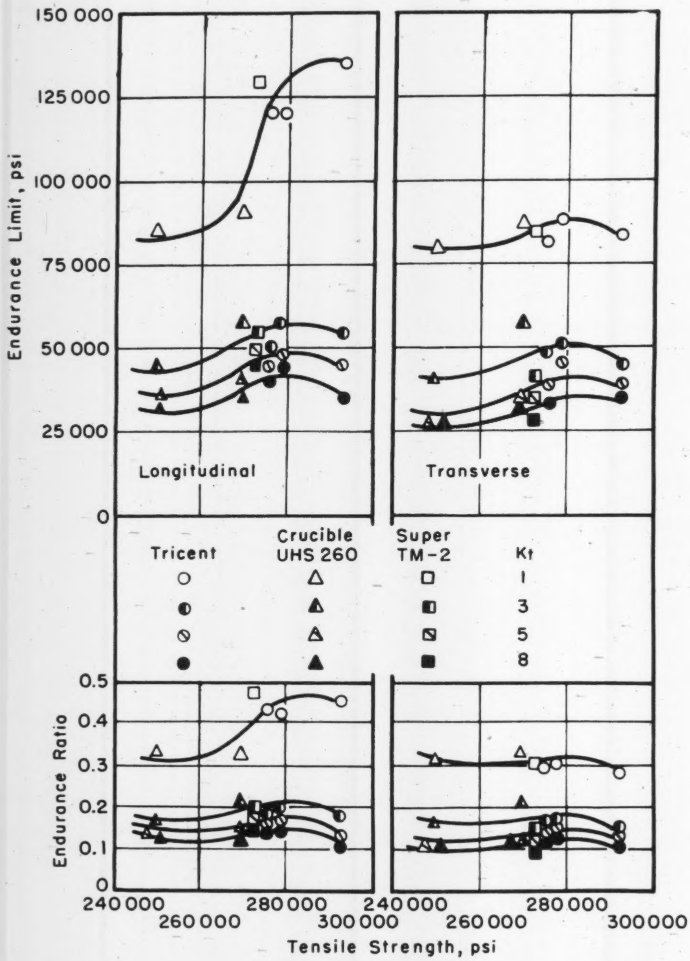


FIG. 10.—Variation of Endurance Limit and Endurance Ratio of High-Strength Steels with the Tensile Strength.

The results presented in Fig. 9 indicate that, of the three steels examined, Tricent steel is the most sensitive to notching, particularly when tempered at 400 F. On tempering at 550 and 700 F, Tricent steel exhibits a notch sensitivity comparable to that of Super TM-2

### *Dependence of Endurance Limit on Tempering Temperature or Strength Level:*

It has been generally accepted that the endurance limit of heat-treated steels increased with increase in strength level to a maximum value at a strength level of approximately 200,000 psi and then gradually decreased with further increase in strength (1). However, the results of tests on a number of high-strength steels (1, 2, 3) have demonstrated that the endurance limit may continue to increase with increase in the strength level above 200,000 psi. In the present investigation, the strength level examined extended between 240,000 and 300,000 psi approximately, and the results obtained were, generally, in good agreement with the trends previously established for rotating-beam tests.

Figure 10 summarizes the results of fatigue tests performed on three steels which ranged in carbon content from 0.35 to 0.41 per cent. This graph illustrates the relationship of the endurance limit and the endurance ratio to the tensile strength. For all testing conditions examined, the endurance limit appears to attain a minimum value at a strength level between 240,000 and approximately 260,000 psi. Above 260,000 psi, the endurance limit increases to a maximum value which occurs between 280,000 and 300,000 psi for smooth specimens and between 270,000 and 290,000 psi for notched specimens.

The endurance ratio "is related to" the tensile strength in a manner similar to that of the endurance limit.

### *Effects of Directionality on the Endurance Limit of High-Strength Steels:*

Figure 10 illustrates the reduction in endurance limit produced by changing the specimen orientation from parallel to perpendicular to the rolling direction. The effect of directionality on the endurance limit is observed to be most severe with unnotched (smooth) specimens. The endurance limit of transverse smooth specimens may be as much as 40 per cent lower than longitudinal smooth specimens depending upon the strength level. This effect is pronounced at high strength levels and decreases with decrease in strength level.

The effect of directionality on the endurance limit of notched specimens is much less severe than that discussed for smooth specimens. However, the effect does exist and is responsible for a maximum drop of about 15 per cent in the endurance limit when the specimen orientation is changed from longitudinal to transverse. The fact that the difference between longitudinal and transverse strength is greater for smooth than for notched specimens is probably explained by a difference in stress state. The regular tension test comprises uniaxial load application, while high lateral stresses develop on loading a notched section. Therefore, in a tension test on a smooth section, only the metal properties in the testing direction are of significance. In contrast, the strength of notched sections may well depend on the properties in the directions perpendicular to that of testing, as well as in that of testing.

## CONCLUSIONS

The results presented in this paper can be summarized as follows:

1. The fatigue strength of notched specimens in the low cycle range may be greater or less than the fatigue strength of smooth specimens depending upon the number of cycles, and notch sensitivity of the steel. In general, the notch-fatigue strength is above the smooth-fatigue strength below about 100 cycles and the reverse is true beyond about 100 cycles.
2. The endurance limit decreases with increase in the stress concentration. This effect is pronounced for stress concentrations between 1 and 3 and decreases with further increase in the stress concentration.

3. In general the endurance limit and the endurance ratio of both smooth and notched specimens develop minimum values between 240,000 and 260,000 psi and maximum values between 270,000 and 300,000 psi.

4. The effects of directionality on the endurance limit are more pronounced with smooth than with notched specimens and at high rather than at low strength levels.

*Acknowledgments:*

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## REFERENCES

(1) G. Sachs, "Survey of Low-Alloy Aircraft Steels Heat-Treated to High-Strength Levels, Part 2—Fatigue," TR 53-254 Wright Air Development Center (1954).

(2) M. A. Melcon, "Ultra High-Strength Steel for Aircraft Structures," *Product Engineering*, Vol. 24, Oct., 1953, p. 129.

(3) G. Sachs, B. B. Muvdi, and E. P. Klier, "Effect of a Number of Variables on the Fatigue Properties of High-Strength Steels," Papers on Metals, Am. Soc. Testing Mats., p. 89 (1956). (Issued as separate publication *ASTM STP No. 196.*)

(4) R. E. Peterson, "Stress Concentration Design Factors," John Wiley and Sons, Inc., New York, N. Y. (1953).

(5) E. C. Hartmann, and G. W. Stickley, "The Direct-Stress Fatigue Strength of 17 ST Aluminum Alloy Through the Range from ½ to 500,000,000 Cycles of Stress," *Technical Note No. 865*, National Advisory Committee for Aeronautics (1942).

(6) M. H. Weisman, and M. H. Kaplan, "The Fatigue Strength of Steel Through the Range From ½ to 30,000 Cycles of Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 649 (1950).

(7) A. Pomp, and M. Hempel, "Notch-Impact Energy, Fatigue Strength and Endurance Limit of Smooth and Notched Specimens of Various Steels Under Axial-Tension Loading," *Archiv Eisenhüttenwesen*, Vol. 21, p. 67 (1950–1951).

# RELATION BETWEEN DIRECT-STRESS AND BENDING FATIGUE OF HIGH-STRENGTH STEELS*

BY GEORGE SACHS[1] AND G. SCHEVEN[2]

## SYNOPSIS

This paper presents the results of fatigue tests on SAE 4340 steel heat treated to several strength levels and tested under various conditions. Direct-stress and rotating-bend tests on smooth and notched specimens were performed leading to failure at ranges between a few and about one million cycles. Each *S-N* curve extrapolates well to the respective static strength as origin. Two characteristic quantities were determined for various cycles: the ratio of the strengths in bending to that in tension (bend-strength ratio) and the ratio of the strengths of notched to smooth specimens (notch-strength ratio). The bend-strength ratio for high numbers of cycles is practically equal to unity, while for low numbers of cycles it is larger than two for smooth specimens and between one and two for notched specimens, depending upon their ductility. The notch-strength ratio is about 0.3 for high numbers of cycles, above 100,000, and then gradually increases with decreasing number of cycles to the static values, which vary greatly with the steel condition. These are also considerably lower for bending than for tension if the steel is notch sensitive.

Fatigue data for small numbers of cycles to failure are becoming increasingly important for a variety of applications. The laws which determine the endurance limit and fatigue strength for high cycle numbers definitely do not apply if the number of cycles is very low, say less than 1000. For example, the high-cycle fatigue strength is generally considered to be practically the same for both rotating bending or tension-compression of equal magnitude ($R = -1$). This applies both to smooth and to notched specimens (**1**).[3] On the other hand, it is generally recognized that the nominal bending strength of ductile materials is considerably higher than the tensile strength (**2**).

A number of tests in this laboratory on heat-treated steel confirmed the fact that the bend-strength ratio, that is, the ratio of strength in bending to strength in tension, is usually higher than unity. This is found to apply not only to smooth specimens, but also to both static and low-cycle fatigue-strength values of notched specimens.

From this scattered evidence it ap-

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 680.

TABLE I.—COMPOSITION AND SIZE OF THE VARIOUS HEATS OF SAE 4340 STEEL.

| Heat | Diameter, in. | Alloying Elements, per cent | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Carbon | Manganese | Phosphorus | Sulfur | Silicon | Nickel | Chromium | Molybdenum |
| No. 1...... | 4 1/4 | 0.41 | 0.79 | 0.013 | 0.016 | 0.31 | 1.83 | 0.77 | 0.23 |
| No. 5...... | 7/8 | 0.39 | 0.76 | 0.015 | 0.015 | 0.29 | 1.84 | 0.72 | 0.23 |
| No. 6...... | 9/16 | 0.39 | 0.71 | 0.017 | 0.022 | 0.30 | 1.87 | 0.94 | 0.24 |

peared interesting to determine the fatigue curves in rotating bending and direct stress (tension-compression) over a wide range of cycles for several materials and to establish general relations between direct-stress and bending-fatigue strength. The low-alloy steel 4340 was selected for this investigation, as a considerable amount of test data was available for various heats of this steel from previous investigations in this laboratory (3–7).

Smooth and sharply-notched specimens of two strength levels, 210,000 psi (800 F tempering temperature) and 290,000 psi (400 F tempering temperature) were systematically investigated at cycles ranging from static tension and bending to 100,000 or higher.

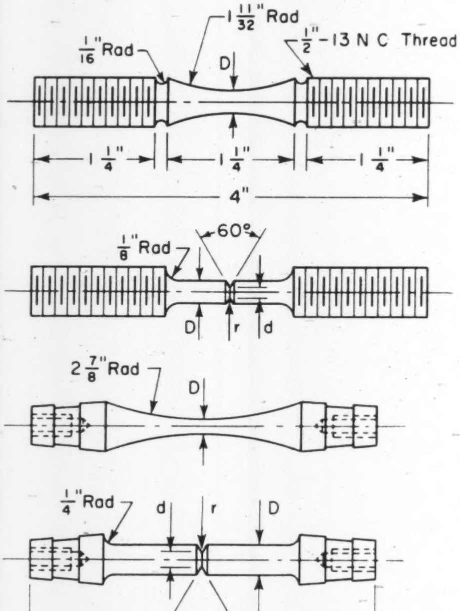
## MATERIAL AND EXPERIMENTAL PROCEDURE

### *Material and Specimens:*

The results reported relate to three heats of the high-strength low-alloy steel SAE 4340. The chemical composition and the section sizes of these steels are given in Table I.

A number of the tests included were obtained on another project which explains the fact that several heats were used. Also the conditions for the various test series differed slightly in several respects. However, the results agreed rather closely, with the exception of one instance which will be discussed later.

The test specimens used are presented in Fig. 1. These were rough machined to about 0.015 in. oversize, heat treated, and then finished to the given dimen-



| Specimen | $D$, in. | $d$, in. | $r$, in. | $K_t$ |
|---|---|---|---|---|
| *a*........... | 0.200 | .... | .... | 1 |
| *b*........... | 0.250 | 0.177 | 0.001 | 7–8 |
| *c*........... | 0.188 | .... | .... | 1 |
| *d*........... | 0.265 | 0.188 | 0.001 | 7 |

Fig. 1.—Dimensions of the Investigated Specimens and Their Notch Concentration Factors $K$.

(*a*) Smooth direct stress fatigue specimen.
(*b*) Notched direct stress fatigue specimen.
(*c*) Smooth rotating beam fatigue specimen.
(*d*) Notched rotating beam fatigue specimen.

sions. Smooth specimens were finish-ground and polished. The notches were finish-machined with a carbide tool. The heat treatment consisted of austenizing at 1600 F for 2 hr, quenching in oil, and tempering for either 1 or 4 hr at a temperature of 400 or 800 F, respectively.

*Testing Procedures:*

Rotating-bending fatigue tests were performed on an R. R. Moore fatigue machine which was geared down to 250 rpm by means of a variable gear and a transmission. In order to obtain cycles to failure in the range up to 15 revolutions, this machine was turned by hand at a rate of approximately 10 rpm.

The data for static bending were obtained by discontinuous loading of this machine in rest. Weights were applied in units of 5 lb, which is equal to approximately 3 per cent of the maximum load. About 2 to 3 min were required for loading. Fracture of notched specimens was observed in all instances. In contrast, smooth specimens did not break but bent plastically to such an extent that further loading was impossible. The respective maximum load applied was taken as the lower limit for the static bending strength. All bending stresses reported are nominal values, calculated according to the theory of elasticity with $E = 30,000,000$ psi.

Direct-stress fatigue data were obtained by means of a Baldwin-Sonntag machine equipped with a direct-stress fixture permitting applications of ±7840 lb. In order to obtain the set load, the machine must run at its full speed of 1800 rpm. When starting, it takes about 5 to 6 sec to come up to speed. Failures occurring within less than 6 sec, therefore, have been discarded. The revolution counter of this machine also registers only every 100 cycles. Because of this it is difficult to obtain accurate data in the range of low-cycle numbers to failure.

The static tension tests were performed on a Baldwin-Lima-Hamilton 60,000-lb tension testing machine. The notched specimens were identical with the direct-stress fatigue specimens. By means of a specially-designed fixture it was possible

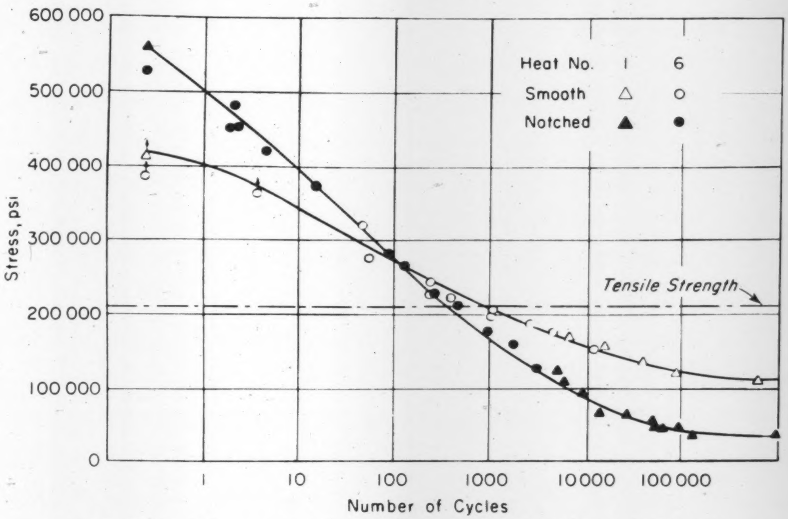


FIG. 2.—*S-N* Curves for Rotating Bending of 210,000-psi, 4340 Steel.

to use these specimens also for static tests.

### CORRELATION BETWEEN THE DIFFERENT TEST PROCEDURES

From the various tests performed, an attempt was made to establish for each of two strength levels, 210,000 and 290,000 psi, four fatigue (*S-N*) curves, as follows:

1. Direct-stress fatigue curves for smooth specimens (stress concentration factor, $K_t = 1$) and for a ratio of lower stress (compression) to upper stress (tension) or stress ratio, $R = -1$.
2. Direct-stress fatigue curves for notched specimens having a $K_t =$ approx mately 8 and for $R = -1$.
3. Rotating-bending fatigue curves for smooth specimens ($K_t = 1$).
4. Rotating-bending fatigue curves for notched specimens having a stress concentration factor, $K_t$, of approximately 7.

These are represented in Figs. 2 to 5.

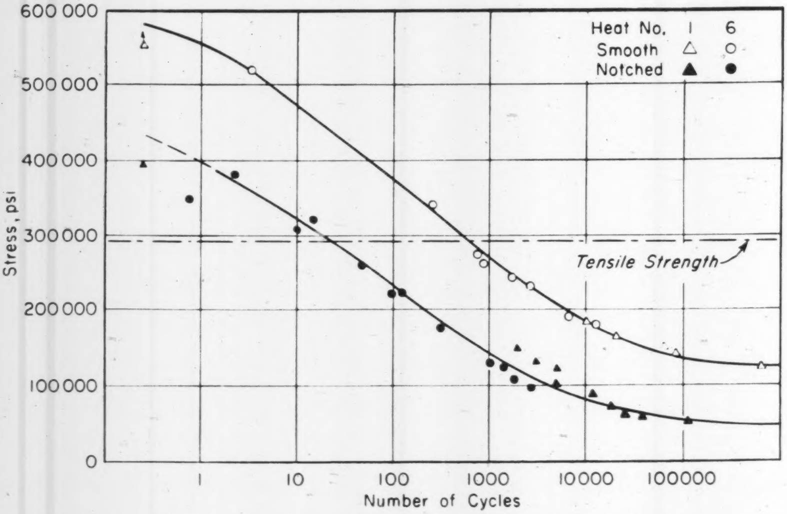


FIG. 3.—*S-N* Curves for Rotating Bending of 290,000-psi, 4340 Steel.

The bending-fatigue curves are based on tests performed at various times with specimens from several heats and section sizes and at different cycling rates. Failures at high stresses and low cycles, up to approximately 10,000, were obtained by means of tests at a cycling rate of 250 rpm. Test data for low stresses and high cycles were added from previous studies of specimens taken from a large section and tested at 10,000 rpm (**4**). In spite of this difference, three of the *S-N* curves match smoothly in their adjoining or overlapping portions. The fourth *S-N* curve reveals some difference in the results of the two series of tests which slightly exceeds the scattering of such data. This discrepancy is of no significance for the conclusions arrived at in this report.

Furthermore, a static test may be considered as the origin of a fatigue curve (**8–11**). Various cycle numbers have been ascribed to the static tests, but it appears that $N = \frac{1}{4}$ is the correct one. Actually, the stress pattern in a static test is equivalent to that in the first $\frac{1}{4}$ of the first cycle of a fatigue test. Again, the rate of testing in the static test was much lower than in the low-cycle fatigue tests performed. However, a separate series of tests at ±200,000 psi has shown that the cycle number obtained at a cycling rate of about 0.3 rpm, which corresponds to a loading rate rather close to that in static tests, and the cycle number obtained at 250 rpm was practically the same, while that at 10,000 rpm was about twice that at 250 rpm.

Figures 2 and 3 confirm that the static bending strength is the origin of the rotating-bending *S-N* curve, and that the effect of variations in testing conditions are within the range of scattering of the test data. In all instances, the bending fatigue strength is found to decrease rather sharply as the number of cycles increases from $N = \frac{1}{4}$ to a few cycles.

Regarding the direct-stress tests, it was impossible to measure definitely cycle numbers of less than about 50. However, direct-stress tests on smooth specimens loaded at 90 to 95 per cent of the tensile strength usually failed after several hundred cycles, while slightly higher loads could not be applied without failure in the range of acceleration at an indefinite load. Therefore, again the concept that the fatigue curves originate in the static (tensile) strength is found to be confirmed by the test data, in spite of the great differences in loading rate, as illustrated in Figs. 4 and 5.

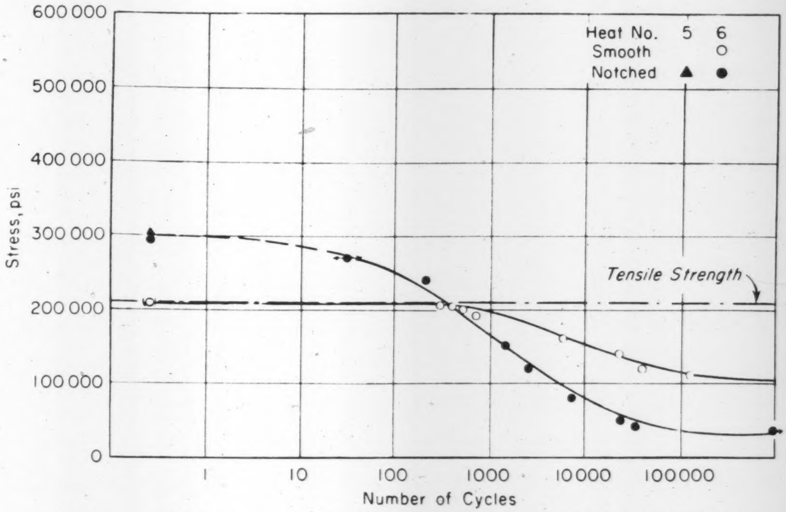


FIG. 4.—*S-N* Curves for Direct Stress ( $R = -1$) of 210,000-psi, 4340 Steel.

The direct-stress fatigue strength for notched specimens was considerably below that of the static test, in the range of high numbers of cycles to failure. However, extrapolation of the *S-N* curves to lower cycles was again in good agreement with the concept that the static (notch) strength is the origin of the *S-N* curve, see Figs. 4 and 5. These were obtained for one or both of the two heats, No. 6 and No. 5 (**3**).

Other factors, such as section size and stress concentration, were reasonably constant within each series (as defined above) of fatigue tests but differed slightly for direct-stress and bending-fatigue tests. The actual test data, however, indicate that these differences are also of little effect on the test data and of no significance for the conclusion drawn from the test data.

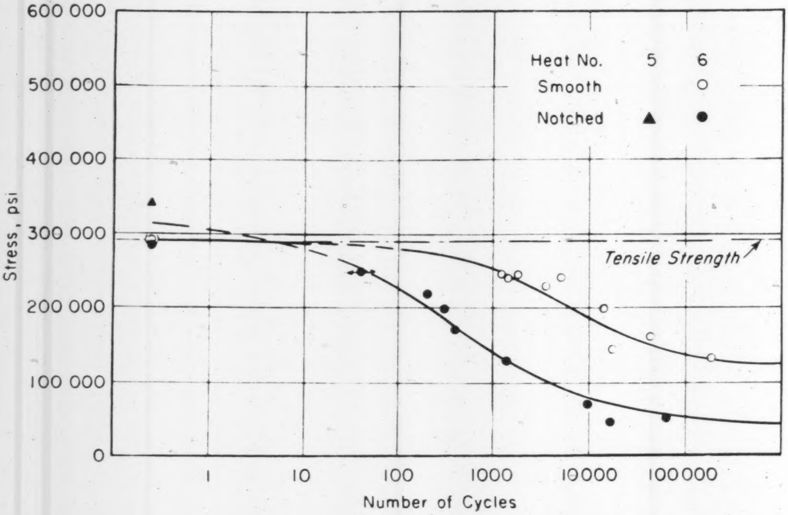


FIG. 5.—*S-N* Curves for Direct Stress ($R = -1$) of 290,000-psi, 4340 Steel.

TABLE II.—EXPERIMENTAL DATA OBTAINED FROM STATIC TESTS.

| Tempering Temperature | 400 F | 500 F | 600 F | 700 F | 800 F |
|---|---|---|---|---|---|
| Ultimate tensile strength, $S_{UT}$, psi | 290 000 | 270 000 | 250 000 | 230 000 | 210 000 |
| Ultimate bending strength, $S_{UB}$, psi | >550 000 | >540 000 | >500 000 | ... | >415 000 |
| Average notch tensile strength, $S_{NT}$, psi | 315 000 | 320 000 | 315 000 | 310 000 | 300 000 |
| Notch bending strength, $S_{NB}$, psi | 395 000 | 485 000 | 525 000 | ... | 560 000 |

## RESULTS OF TESTS

### *Static-Strength Relations:*

The experimental data obtained from static tests are assembled in Table II. Included in this table are the results of tests on several additional strength levels, obtained by tempering at 500, 600, and 700 F respectively. As explained above, the bending strength of smooth specimens could not be determined, because of excessive plastic deformation. Therefore, the values reported are those measured at the maximum deformation. These values are found to be approximately equal to twice the tensile strength; therefore, for the further analysis, the bending strength of smooth specimens is simply taken as twice the tensile strength. The static notch strengths in this table, which are used for the evaluation of the notch-strength ratio, $R_{NT}$, are averages of the values for the two heats No. 5 and No. 6 in the case of the 210,000 psi and 290,000 psi strength levels. In the other instances the values for heat No. 5 were slightly reduced according to the above disclosed

TABLE III.—RATIOS OF STRENGTH VALUES GIVEN IN TABLE II.

| Tempering Temperature | 400 F | 500 F | 600 F | 700 F | 800 F |
|---|---|---|---|---|---|
| Notch-strength ratio for tension, $R_{NT}$ | 1.08 | 1.18 | 1.26 | 1.35 | 1.43 |
| Notch-strength ratio for bending, $R_{NB}$ | <0.72 | <0.90 | <1.05 | ... | <1.35 |
| Bend-strength ratio for smooth specimen, $R_{BS}$ | >1.90 | >2.00 | >2.00 | ... | >1.98 |
| Bend-strength ratio for notched specimen, $R_{BN}$ | 1.25 | 1.51 | 1.67 | ... | 1.87 |

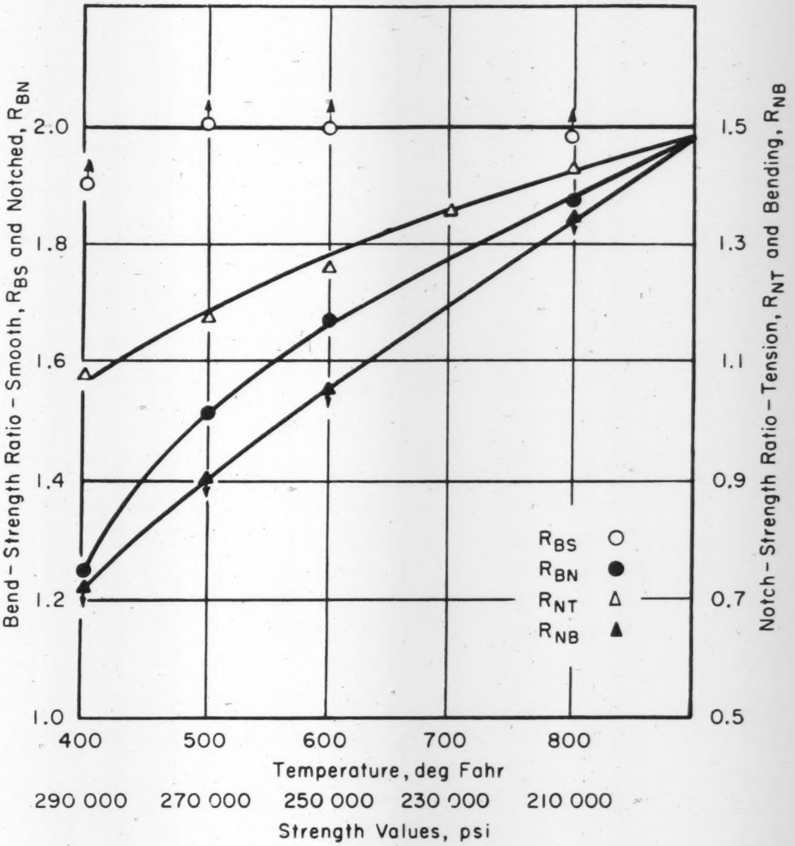


FIG. 6.—Relation Between Static Strength Ratios and Tempering Temperature or Strength Level.

differences between the two heats. The individual values are included in Figs. 4 and 5. The static tension tests were not repeated, because these data were already obtained in a previous series where specimens of heat No. 6 of the same dimensions and same heat treatment were investigated (5).

In Table III the following ratios of the values given in Table II are assembled:

$R_{BS}$ (bend-strength ratio for smooth specimens) = ratio of ultimate bending strength ($S_{UB}$) to ultimate tensile strength ($S_{UT}$) for smooth specimens.

$R_{BN}$ (bend-strength ratio for notched specimens) = ratio of notch-bending strength ($S_{NB}$) to notch-tensile strength ($S_{NT}$).

$R_{NT}$ (notch-strength ratio for tension test) = ratio of notch-tensile strength ($S_{NT}$) to ultimate tensile strength ($S_{UT}$).

$R_{NB}$ (notch-strength ratio for bend test) = ratio of notch-bending strength ($S_{NB}$) to ultimate bending strength ($S_{UB}$).

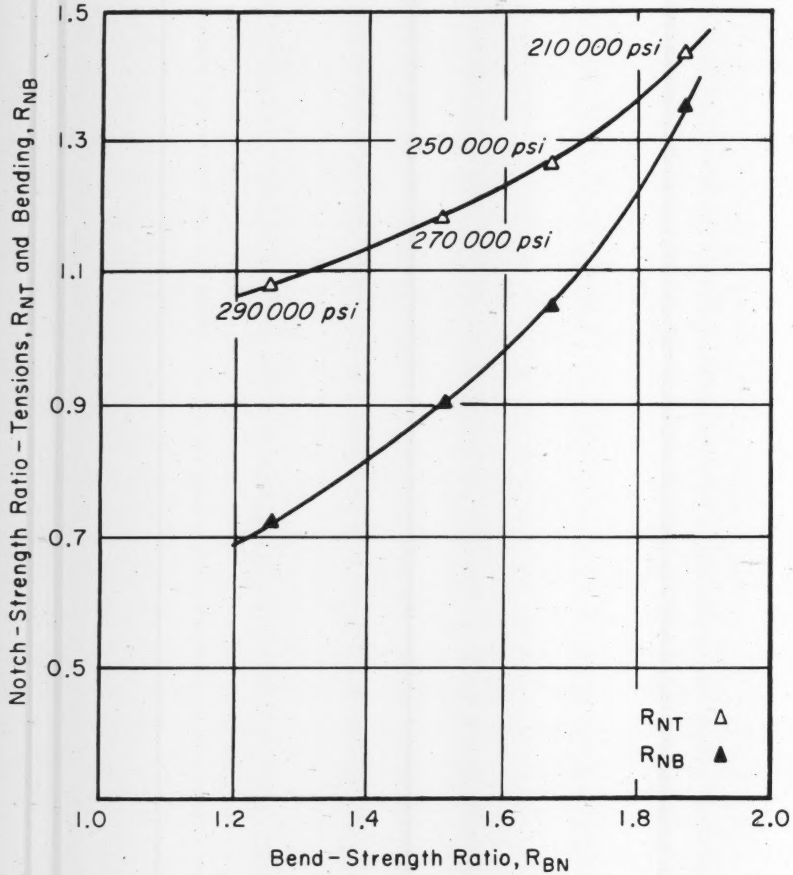


FIG. 7.—Relation Between Notch-Strength Ratio for Tension and Bending *versus* Bend-Strength Ratio for Notched Specimens at Various Strength Levels.

| | | |
|---|---|---|
| 290,000 psi | Strength Level | 210,000 psi |
| 400 F | Tempering Temperature | 800 F |

The fact that the bend-strength ratio for smooth specimens is approximately two, or perhaps even higher, is in conformance with the fact that the ductility of all these steel conditions is high (2). The reduction of area in tension of these steels is between 45 and 50 per cent, and their ductility in bending is also rather large.

The bend-strength ratio of notched specimens is, according to Table III, less than two and rather different for the various strength levels. It decreases rapidly with increasing strength level, as further illustrated in Fig. 6. Therefore, it may be assumed that the ductility of the steel, in the presence of stress concentrations, also decreases considerably as the strength level increases. The minimum value observed here is about $R_{BN} = 1.2$, or rather close to that of the completely brittle condition which should be unity (2). This agrees with the fact that notched specimens of high-strength low-alloy steel always break in an apparently brittle manner (3–7, 12, 13). How to evaluate their low ductility is not clarified; it is a matter of considerable concern to designers.

In previous investigations on high-strength steels, the notch-strength ratio in tension was found to be a reliable measure of their ductility in the presence of high stress concentrations. For the sharply-notched specimen used, a notch-strength ratio of about 0.4 appears to indicate a maximum condition of brittleness obtainable in this group of materials. SAE 4340 steel, when heat treated to strength levels up to 290,000 psi, exhibits considerably higher notch-strength ratios (in small specimens) anywhere between 1.0 and the maximum value of 1.5 of notch-ductile steels. According to Table III and Fig. 6, the notch-strength ratio in tension ($R_{NT}$) increases as the tensile strength decreases, indicative of a simultaneous increase in ductility.

Values of the notch-strength ratio in bending are apparently reported here for the first time. They are found, according to Table III and Fig. 6, to vary considerably more than the notch-strength ratio in tension, namely, between 0.7 and almost 1.5. The latter value again appears to be the possible maximum for a notch-ductile steel condition and the specimen shape used in these tests.

In Fig. 7 the notch-strength ratios for both tension and bending are plotted *versus* the bend-strength ratio of notched specimens. This graph discloses a rather definite relation between notch-strength ratio and bend-strength ratio. However, the latter approaches its minimum value of unity long before the notch-strength ratio in tension indicates the existence of an excessively brittle condition, characterized by a value considerably below 1.0. The meaning of this relation is that the notch-strength ratio appears to be rather sensitive to differences in small values of ductility. In contrast, Fig. 6 leads to the conclusion that all notch-brittle steel conditions possess a bend-strength ratio near 1.0. Therefore, this quantity is not suitable for differentiating such steels.

Another interesting conclusion which may be deduced from these studies is that steels may be classified as rather brittle in the presence of high stress concentrations from their performance in either tension or bending. There is considerable doubt whether the surface ductility of a notched specimen which has a low average (notch) ductility is actually very small (14). However, whatever the value of surface ductility may be, it appears that the average ductility and notch strength are much more important material characteristics in regard to the load-carrying capacity of metal structures in the presence of stress concentrations.

The finding that the notch-strength ratio is lower in bending than in tension by an amount that increases as the notch-strength value decreases (Fig. 6) is actually predetermined by the above-discussed relations between notch-strength ratio in tension, $R_{NT} = \frac{S_{NT}}{S_{UT}}$; in bending, $R_{NB} = \frac{S_{NB}}{S_{UB}}$; and bend-strength ratio of notched specimens, $R_{BN} = \frac{S_{NB}}{S_{NT}}$. As the bend strength for smooth specimens, $R_{BS} = \frac{S_{UB}}{S_{UT}}$, is usually about 2, the following formula applies:

$$R_{NB} = \frac{S_{NB}}{S_{UB}} = \frac{S_{NT}}{S_{UT}} \times \frac{S_{NB}}{S_{NT}} \times \frac{S_{UT}}{S_{UB}}$$

$$= R_{NT} \times \frac{R_{BN}}{R_{BS}}$$

$$= \text{approximately } R_{NT} \times \frac{R_{BN}}{2}$$

Therefore, the one limit of high ductility, where $R_{NT} = 1.5$ and $R_{BN} = 2$, yields:

$$R_{NB} = R_{NT} = 1.5$$

The other limit of rather low ductility, where $R_{BN} = 1$, yields:

$$R_{NB} = 0.5\, R_{NT}$$

*Fatigue Strength at Various Cycle Numbers:*

The four *S-N* curves obtained for each of the two strength levels of the 4340 steel are presented in Figs. 2 to 5. These curves are replotted on a small scale in Figs. 8(*a–h*). Each of these latter graphs compares two of the fatigue curves for one steel, which agree in one and differ in the other of the two variables: namely, stress concentration, $K_t = 1$ and $K_t = 7$ respectively; and stress application, direct stress and rotating bending, respectively.

These tests were systematically conducted only up to about 100,000 cycles with emphasis placed on tests yielding low numbers of cycles. However, from previous tests it appears established that the relative position of different curves does not materially change as the cycle number increases above 100,000 (**15**).

For the purpose of comparing the *S-N* curves obtained under different testing conditions, these may be subdivided into two portions, namely, the low-cycle range between the static test and approximately $N = 10{,}000$ and the high-cycle range at cycle numbers greater than $N = 10{,}000$.

In the high-cycle range, the relations between each set of curves are very simple. The test data for bending and direct stress (**1**) are then found to be practically identical. This applies both to the smooth specimens, Figs. 8(*e–h*), and to the notched specimens.

Regarding the effect of notching in the high-cycle range it is observed, in confirmation of previous evidence (**15**), that the ratio between smooth- and notch-fatigue strength for a given cycle number, say above 10,000, is nearly independent of this number of cycles.

This ratio, which is the reciprocal value of the notch-strength ratio, may be termed "retained stress concentration factor." If the initial (elastic) stress concentration factor is high, its retained value is generally considerably lower. The retained stress concentration factor also depends upon the steel composition and condition. It may vary between unity (that is complete elimination of the stress concentration) and a maximum value of about four (**7**).

It may be mentioned that in a number of instances previous test data indicated slightly higher retained stress concentrations at 10,000 and 100,000 cycles than at one million (**7**). Further tests will be necessary to establish more definitely this fact.

The low-cycle portions of the fatigue curves need little further discussion. In all instances, the *S-N* curve gradually approaches and extrapolates to the static strength as the number of cycles decreases to small values. However, in the case of the investigated high-strength steels, the fatigue strength in rotating bending of both smooth and notched specimens is clearly lower than the static strength, even for a few cycles. In contrast, the low-cycle fatigue strength of smooth specimens in direct stress changes only to an insignificant extent as the number of cycles varies between $\frac{1}{4}$ to several hundred cycles. Finally, the direct-stress fatigue strength of notched specimens also distinctly decreases in the range of less than 100 cycles.

The above-introduced concept that both the bend-strength ratio and the notch-strength ratio measure, in some complicated fashion, the ductility of the material can also be applied to the change of these quantities with increasing number of cycles. These can be determined from the curves in Figs. 9(*a*) and (*b*), but with rather limited accuracy.

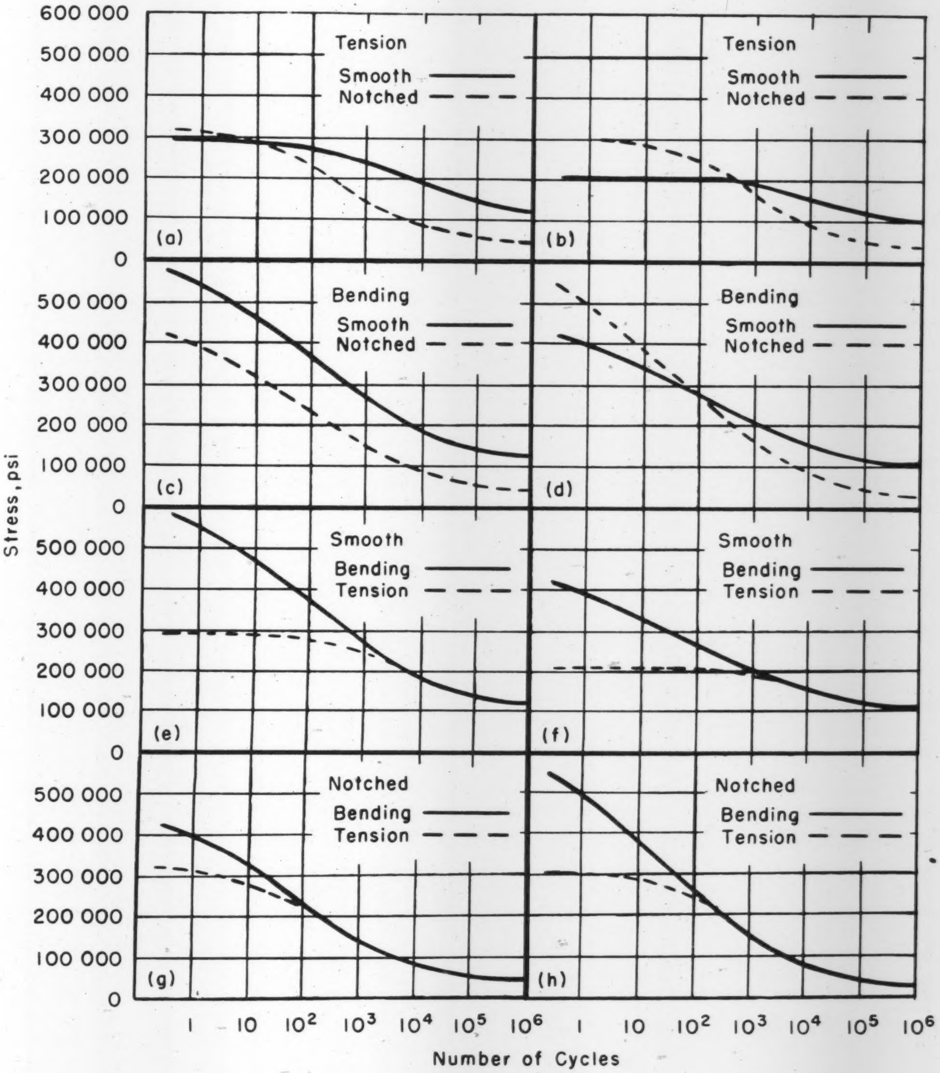


FIG. 8.—Comparison of *S-N* Curves for 4340 Steel Under Various Testing Conditions.

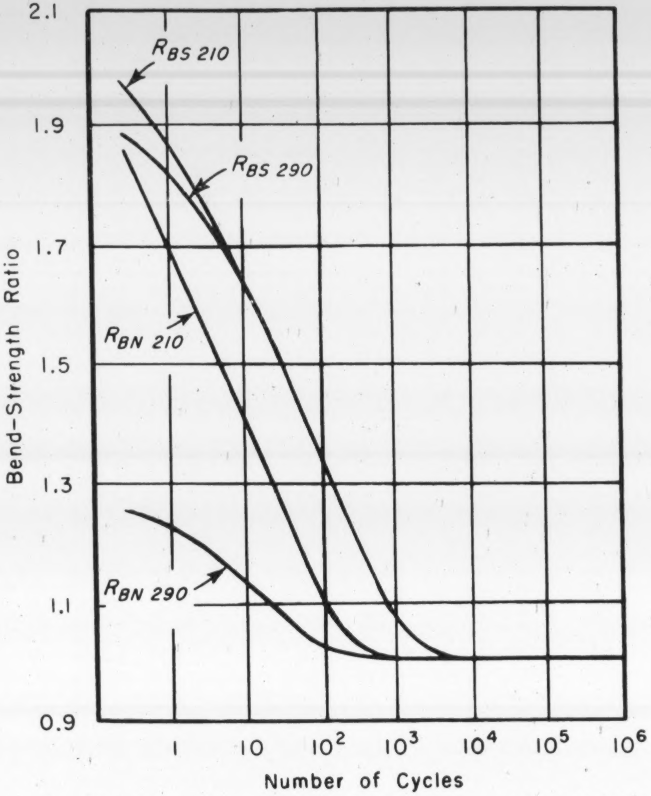


FIG. 9(*a*).—Bend-Strength Ratio for Smooth and Notched Specimens at two Strength Levels *versus* Cycles to Failure.

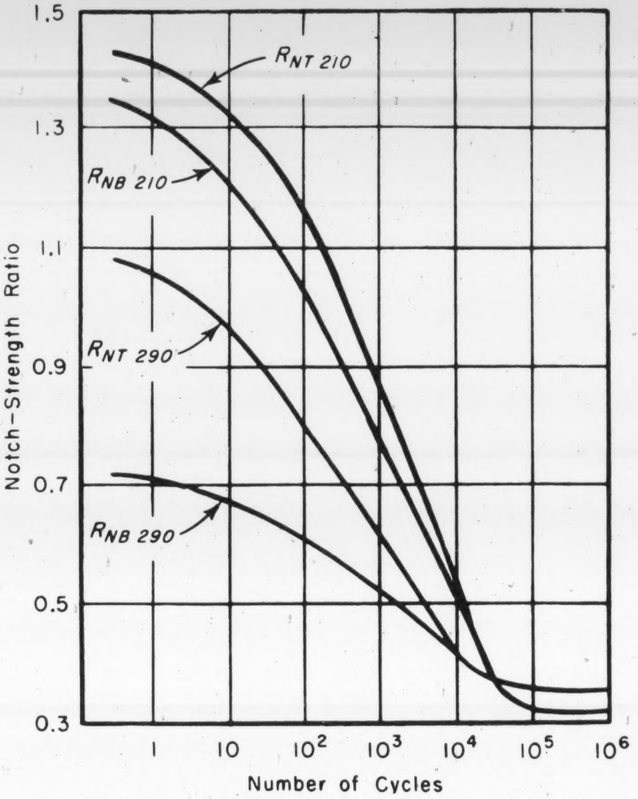


FIG. 9(*b*).—Notch-Strength Ratio for Tension and Bending at two Strength Levels *versus* Cycles to Failure.

Figure 9(*a*) presents the curves for the bend-strength ratios for both smooth and notched specimens and for both the 210,000 psi and 290,000 psi strength levels. Figure 9(*b*) similarly shows the four curves for the notch-strength ratios for both tension and bending and for both the 210,000 psi and the 290,000 psi tempers.

It can be seen from Fig. 9(*a*) that, in all instances, the bend-strength ratio decreases first rather slowly, then rapidly, and finally again very slowly, from its initial static value to the final minimum value of unity, as the number of cycles increases. The limiting value is generally reached already at cycle numbers above 1000. This may be explained by the generally accepted concept that the ductility of a material that fails in high-cycle fatigue is small.

Figure 9(*a*) indicates that the ductile condition in static tension gradually changes into a brittle condition. The bend-strength ratio does not distinguish between the conditions at various numbers of cycles exceeding 1000. As discussed above, it may be suspected that the bend-strength ratio again does not respond to variations in small ductility values, while the notch-strength ratio is found to be more sensitive in this respect.

Indeed, Fig. 9(*b*) reveals that the notch-strength ratio assumes its minimum value of about 0.3 for both steel conditions, as well as for tension and bending, only at cycles over 100,000. At 1000 cycles to failure the notch-strength ratio is found to be considerably higher and roughly half way between the limiting values. These findings indicate that the embrittlement by repeated cycles is not complete unless the number of cycles at failure exceeds 100,000.

## DISCUSSION AND CONCLUSIONS

It is evident from the test data that the relations which determine the endurance limit and the fatigue strength at high numbers of cycles are fundamentally different from those at low numbers of cycles.

The high-cycle range, in this respect, extends down to approximately 10,000 cycles or less. In this range, a certain initial stress-concentration effect is fully or partly retained, depending upon its initial magnitude and the metal characteristics. The metal performs in a rather brittle manner, under such circumstances.

The ratio between bending and direct-stress fatigue strength is then also found to be practically unity in accordance with the concept that complete brittleness and perfect elasticity are two facets of the same phenomenon. It appears from the tests, therefore, that the materials tested must be considered sufficiently ductile in fatigue tests to relieve considerably a high stress-concentration effect at comparatively low stresses. On the other hand, such small values of ductility seem to have less effect on the bend-strength ratio.

As the number of cycles to failure decreases progressively below 100,000 and approaches few cycles, a rather gradual transition to the relations governing static tension and bending occurs. In static tests on most materials an initial stress concentration is usually relieved to a considerably greater extent than in fatigue tests. Exceptions, represented by extremely notch-brittle steels (**13**) may exist, but such steels have not yet been studied.

The static bend-strength of smooth specimens indicates that these possess sufficient ductility to lead to maximum bend-strength values. On the other hand, sharply-notched specimens of the steels investigated remain subject to some notch sensitivity, or a retained stress-concentration effect, as indicated by their notch-strength values being less than 1.5. The magnitude of this effect greatly depends upon the steel condition.

According to Fig. 6 the bend-strength ratio changes in proportion to the notch-strength ratio. However, if the notch-strength ratio in tension approaches unity, which is indicative of a still rather moderate stress-concentration effect, the bend-strength ratio already reached its theoretically possible minimum of unity. It might again be interesting to study more notch-sensitive steels in this respect.

The validity of the calculations, such as presented above, depends upon the assumption that the metal possesses equal ductility in tension and in bending, on the one hand, and particularly in notch tension and notch bending, on the other hand, other conditions being identical. Considering the fact that the ductility and notch strength of these steels depend upon the section size (**14**) this would then mean that, for any particular section size, the results of tension tests are applicable to the performance of the metal in bending. Further experimental evidence in this respect is necessary. It would also be of considerable fundamental value as it would permit advancing the theory of section-size effects.

*Acknowledgment:*

The authors are indebted to K. Tong for valuable advice, to R. Bale for performance of part of the experimental work, and to B. B. Muvdi for assistance in the selection of materials and procedures.

## REFERENCES

**(1)** G. Sachs, "Survey of Low Alloy Aircraft Steels Heat Treated to High-Strength Levels, Part 2: Fatigue," *TR 53-254* Part 2, Wright Air Development Center (1954).

**(2)** J. M. Frankland and R. E. Roach, "Strength Under Combined Tension and Bending in the Plastic Range," *Journal Aeronautical Science,* Vol. 21, No. 7, p. 449 (1954).

**(3)** G. Sachs, V. Weiss and E. P. Klier, "Effects of Heat Treating and Testing Variables on the Notch-Strength of 4340 Steel," *Proceedings,* Am. Soc. Testing Mats., Vol. 56, p. 649 (1956).

**(4)** G. Sachs, B. B. Muvdi and E. P. Klier, "Design Properties of High-Strength Steels in the Presence of Stress-Concentrations," *TR 55-103,* Supplement 1, Wright Air Development Center (1956).

**(5)** G. Sachs, B. B. Muvdi and E. P. Klier, "Design Properties of High-Strength Steels in the Presence of Stress Concentrations and Hydrogen Embrittlement," *TR 55-18,* Supplement 1, Wright Air Development Center (1955).

**(6)** B. B. Muvdi, G. Sachs and E. P. Klier, "Design Properties of High-Strength Steels in the Presence of Stress Concentrations, Part 1: Dependence of Notch-Tension Properties of High-Strength Steels on a Number of Factors," *TR 56-395,* Part 1 Wright Air Development Center (1956).

**(7)** B. B. Muvdi, G. Sachs and E. P. Klier, "Design Properties of High-Strength Steels in the Presence of Stress Concentrations, Part 2: Axial-Load Fatigue Properties of High-Strength Steels," *TR 56-395,* Part 2 Wright Air Development Center (1956).

**(8)** E. C. Hartmann and G. W. Stickley, "The Direct-Stress Fatigue Strength of 17 ST Aluminum Alloy Through the Range from ½ to 500,000,000 Cycles of Stress," *Technical Note 865,* Nat. Advisory Committee Aeronautics (1942).

**(9)** S. I. Liu, J. J. Lynch, E. J. Ripling and G. Sachs, "Low Cycle Fatigue of the Aluminum Alloy 24 ST in Direct Stress," *Transactions,* Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Div., p. 469 (1948).

**(10)** M. H. Weissman and M. H. Kaplan, "The Fatigue Strength of Steel Through the Range from ½ to 30,000 Cycles of Stress, *Proceedings,* Am. Soc. Testing Mats., Vol. 50, p. 649 (1950).

**(11)** A. Pomp and M. Hempel, "Notch-Impact Energy, Fatigue Strength, and Endurance

Limit of Smooth and Notched Specimens of Various Steels Under Axial-Tension Loading," *Archiv Eisenhuettenwesen*, Vol. 21, p. 67 (1950–1951).

(12) G. Sachs, L. J. Ebert and W. F. Brown, Jr., "Comparison of Various Structural Alloy Steels by Means of the Static Notch-Bar Tensile Test, *Technical Publication 2110*, Am. Inst. Mining Metallurgical Engrs. (1946).

(13) G. Sachs, "Survey of Low Alloy Aircraft Steels Heat Treated to High-Strength Levels, Part 5: Mechanical Properties in the Presence of Stress Concentrations," *TR 53-254*, Part 5, Wright Air Development Center (1954).

(14) M. L. Fried and G. Sachs, "Notched Bar Tension Tests on Annealed Carbon Steel Specimens of Various Sizes and Contours," Symposium on Deformation of Metals as Related to Forming and Service, p. 83 Am. Soc. Testing Mats., (1949). (Issued as separate publication *ASTM STP No. 87*.)

(15) M. A. Melcon, "Ultra High Strength Steel for Aircraft Structures," *Product Engineering*, Vol. 24, Oct., 1953, p. 129.

# EFFECTS OF HYDROGEN ON LOW-CYCLE FATIGUE OF HIGH-STRENGTH STEELS*

By G. Scheven,[1] G. Sachs,[1] and K. Tong[1]

## Synopsis

This report presents the results of low-cycle fatigue tests on SAE 4340 steel, heat treated to strength levels between 210,000 and 290,000 psi. Rotating-bend tests were performed on notched and smooth specimens, in both the as-heat-treated and hydrogen-embrittled conditions. The influence of hydrogen, introduced by cadmium plating, on the fatigue life and crack propagation was investigated, covering a range from approximately 5 to 10,000 cycles.

The hydrogen effect was found to be rather inconsistent. However, the fatigue life was usually reduced, particularly if the strength level was 230,000 to 270,000 psi and if the cycling rate was low. Fracture appearance and crack propagation were greatly influenced by the presence of hydrogen.

The presence of hydrogen in steel is known to cause a pronounced loss in ductility, and, in the presence of stress concentrations, also in strength (**1**)[2] This hydrogen embrittlement has been extensively studied in the laboratory, particularly for super-high-strength steels (**1, 2, 3, 4**). It was found that either as the rate of loading or the magnitude of an applied static ("sustained") load decreases, hydrogen embrittlement becomes very pronounced at very high strength levels, and in the presence of such hydrogen as is introduced by pickling, cathodic cleaning, or plating.

On the other hand, little attention has been paid to the effect of hydrogen on the fatigue properties of steels (**5**). This may be partly due to the fact that the endurance limit of steels is usually observed to be lower than the strength values observed in "sustained loading" or "static fatigue" within equal total times and otherwise similar conditions. Therefore, it appears that if hydrogen embrittlement affects fatigue strength and fatigue life, this would apply particularly (*a*) to low-cycle fatigue at comparatively high stresses, and (*b*) to rather slow repetitions of such stresses. Considering the increasing importance of low-cycle fatigue as a limiting design factor in various applications, it was felt that an investigation of hydrogen embrittlement under such conditions would be of considerable interest.

This preliminary study of the effects of hydrogen concerns its effect on the

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 697.

*S-N* curve, as well as on the propagation of the fatigue crack in notched specimens. A low-alloy steel, SAE 4340, heat treated to strength levels between 210,000 and 290,000 psi, was selected for the investigation. These steel conditions are the same as those used for the fatigue tests described in a previous paper (6).

## MATERIAL AND EXPERIMENTAL PROCEDURE

### *Material and Specimens:*

The results presented in this report relate to two heats of SAE 4340 steel. The chemical composition and the section sizes of these steels are given in Table I.

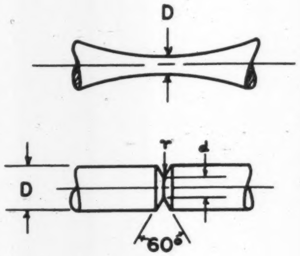
A small number of tests included were obtained on another project. This explains the fact that the results for two heats of 4340 steel are reported. The differences of the test results, however, are within test accuracy and of no influence on the conclusions arrived at in this study.

The dimensions of the test specimens are presented in Fig. 1. These were rough machined to about 0.015 in. oversize, heat treated, and then finish ground to the given dimensions. Smooth specimens were polished after finish grinding, notched specimens were finish machined with a properly ground carbide tool. The heat treatment consisted of austenizing at 1600 F for 2 hr, quenching in oil, and tempering for either 1 or 4 hr at temperatures ranging from 400 to 800 F respectively. The resulting strength levels were as follows:

| | |
|---|---|
| 400 F | 290,000 psi |
| 500 F | 270,000 psi |
| 600 F | 250,000 psi |
| 700 F | 230,000 psi |
| 800 F | 210,000 psi |

TABLE I.—COMPOSITION AND SIZE OF THE VARIOUS HEATS OF SAE 4340 STEEL.

| Heat | Diamter, in. | Alloying Elements of 4340 Steel, per cent | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Carbon | Manganese | Phosphorus | Sulfur | Silicon | Nickel | Chromium | Molybdenum |
| 1 | 4¼ | 0.41 | 0.79 | 0.013 | 0.016 | 0.31 | 1.83 | 0.77 | 0.23 |
| 6 | 9⁄16 | 0.39 | 0.71 | 0.017 | 0.22 | 0.30 | 1.87 | 0.94 | 0.24 |



*Top:*
Smooth Rotating-Beam Fatigue Specimen
*Bottom:*
Notched Rotating-Beam Fatigue Specimen

| Specimen, | *D*, in. | *d*, in. | *r*, in. | *K* |
|---|---|---|---|---|
| Smooth | 0.188 | ..... | ..... | 1 |
| Notched | 0.265 | 0.188 | 0.001 | 7 |

FIG. 1.—Dimensions of the Investigated Specimens and Their Notch Concentration Factors, *K*.

### *Testing Procedure:*

Rotating-bending tests were performed on R. R. Moore fatigue machines which were geared down to speeds ranging from 45 to 2500 rpm.

The investigation on the influence of the strain rate was carried out with a worm gear, in order to obtain testing speeds in the magnitude of 0.33 rpm.

A special device was designed to measure the deflection of the specimen during rotating. The device consisted of a

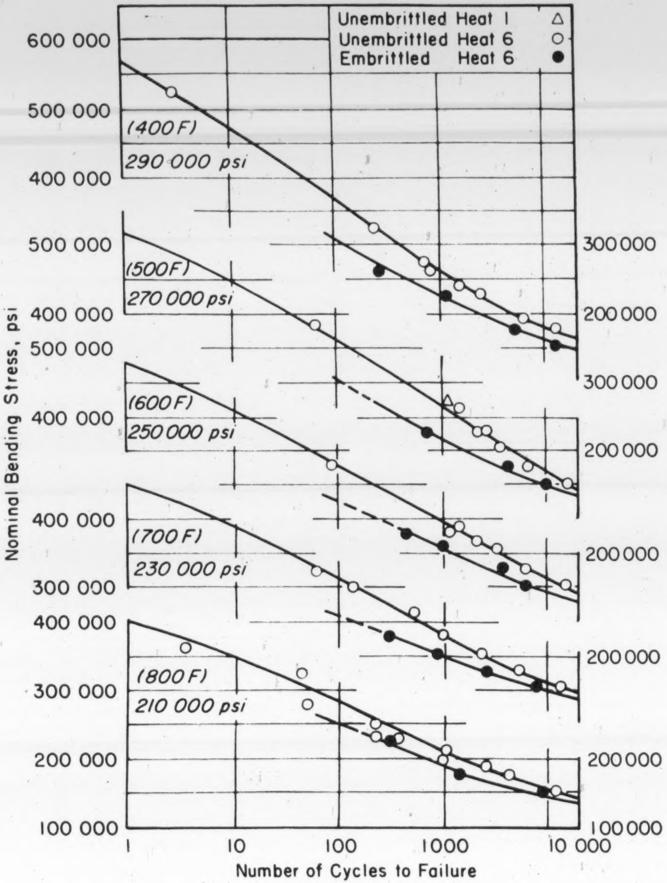


Fig. 2.—*S-N* Curves for Smooth Unembrittled and Embrittled Specimens Tempered to Various Strength Levels.

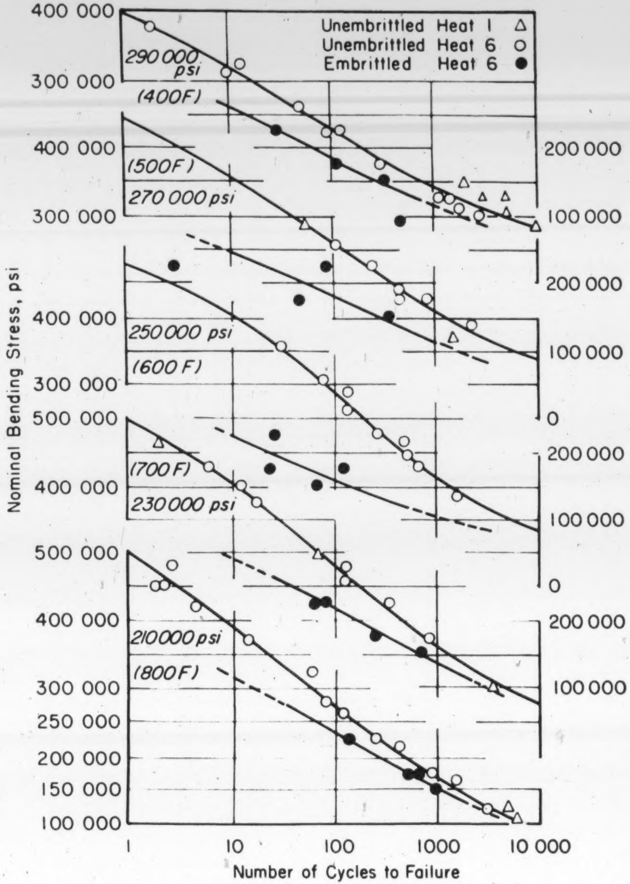


Fig. 3.—*S-N* Curves for Notched Unembrittled and Embrittled Specimens Tempered to Various Strength Levels.

differential transformer and an X-Y recorder. The differential transformer permitted measuring of deflection up to 1 in.

*Plating Procedure:*

The cadmium plating of one series of smooth and notched specimens was carried out by a commercial plater. The bath consisted of:

| | |
|---|---|
| cadmium oxide....... | 3 oz per gal distilled water |
| sodium hydroxide..... | 2.6 oz per gal distilled water |
| sodium cyanide....... | 16.4 oz per gal distilled water |

The plating was performed at room temperature with a current density of 110 ma per sq in. for approximately 20 min, leading to a deposit thickness of approximately 0.0003 in. The specimens were stored after plating in a solution of dry ice and acetone, having a temperature of approximately −75 to −80 C. The plated specimens were stored for about 1 hr at room temperature in order to assume this temperature before the test was started.

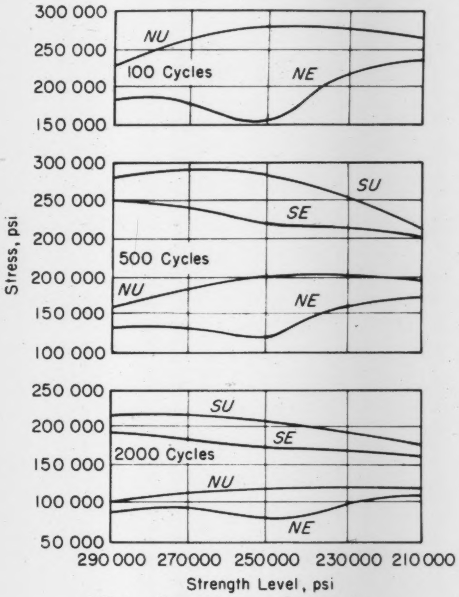
The plating of the notched specimens used in the investigation on the effect of the strain rate was carried out in the laboratory. The composition of the plating bath was:

| | |
|---|---|
| sodium cyanide....... | 16 oz per gal distilled water |
| cadmium oxide....... | 4 oz per gal distilled water |

The plating was performed at a current density of approximately 200 ma per sq in. for 6 min. The bath temperature was approximately equal to the room temperature and changing with the latter. The thickness of the deposit was about 0.0003 to 0.0004 in. The tests were performed instantly after plating.

## RESULTS OF TESTS

In order to disclose an effect of hydrogen embrittlement on the *S-N* curve, it was thought advisable to reduce the speed of one R. R. Moore machine from 10,000 to 250 rpm, both in this investigation and in some previous preliminary tests (5). This was a fortunate decision, as later tests (Fig. 6) indicated that the hydrogen effect completely faded out at very high speeds, and that even at 250 rpm, only rather severe hydrogen embrittlement was revealed by the fatigue tests. Also, apparently the same plating procedure yielded widely different results at different times.



SU = Smooth unembrittled
NU = Notched unembrittled
SE = Smooth embrittled
NE = Notched embrittled

FIG. 4.—Fatigue Strength of Smooth and Notched Specimens in Unembrittled and Embrittled Condition at Various Number of Cycles *versus* Strength Level (at 100, 500 and 2000 Cycles to Failure).

As a consequence, only the first series of commercially plated specimens led to a hydrogen effect that amounted to a multiple of the normal scattering and permitted to establish *S-N* curves for hydrogen embrittled specimens. In the preliminary tests (5) also, S-N curves were obtained, after cathodic treatment of similar steel conditions, which differed from those of the as-heat-treated steel. In this case, the effect amounted roughly to one half of that developed in the present tests, but it was difficult to establish it quantitatively because of the generally large scattering of such data. Therefore, only the results of the one, above-mentioned, series of tests will be discussed in detail.

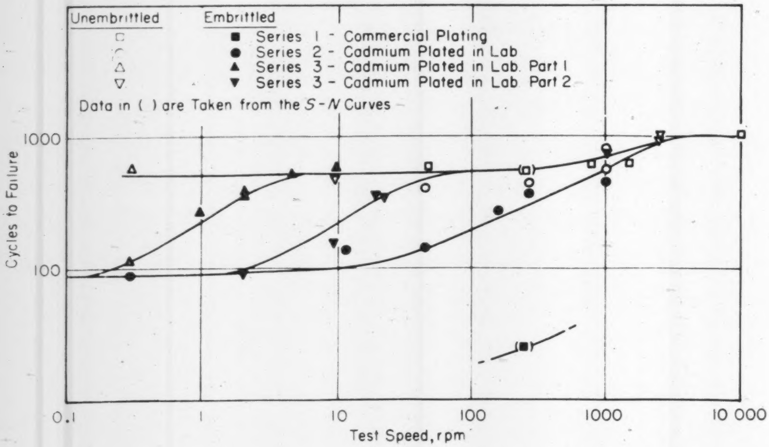


FIG. 5.—Number of Cycles to Failure of Notched Unembrittled and Embrittled Specimens Tempered at 600 F (250,000 psi Strength Level) and Tested at ±200,000 psi Stress *versus* Strain Rate (Test Speed).

*Effect of Hydrogen on the S-N Curve:*

The bending-fatigue data assembled in Fig. 2 for smooth specimens and in Fig. 3 for notched specimens, related to two types of metal conditions: (*a*) as-heat-treated and ground or machined surface, and (*b*) cadmium plated after heat treating and finishing. The governing hydrogen embrittlement are revealed:

1. In all instances this effect decreases as the number of cycles increases and gradually fades out at cycles of about 10,000. This decrease of hydrogen embrittlement with decreasing applied load, is also apparent from Fig. 4, which illustrates the stresses necessary to cause failure at three different cycle numbers: 100, 500, and 2000.

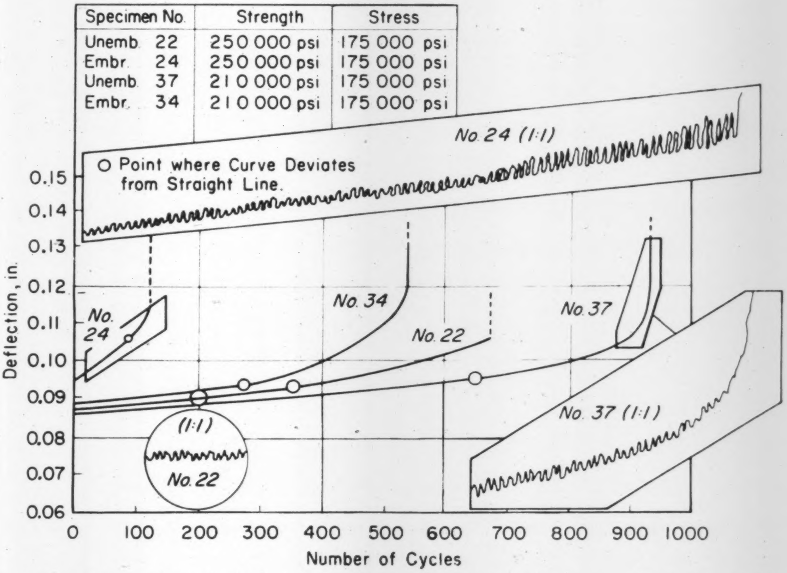
2. In general, the relative effect appears to be considerably larger for notched than for smooth specimens, see Figs. 2 and 3.

3. Regarding the effect of strength level, the greatest reduction in cycles to failure is observed after heat treating to a strength level of 250,000 psi (Figs. 2 to 4). When heat treated to strength levels of 290,000, 270,000, and 230,000 psi, the effect of hydrogen is rather small.

*Effect of Cycling Rate on Hydrogen Embrittlement:*

As already mentioned, the displacement of the S-N curve by plating to lower stress and cycle values was found to be rather inconsistent. In several series of bending-fatigue tests, at the selected rate of 250 rpm, only insignificant differences between unembrittled and embrittled specimens occurred, under otherwise identical conditions.

As hydrogen embrittlement develops with the rate or time of loading during static tests, it was expected that this would apply also to fatigue tests. Therefore, a number of tests on notched specimens were performed at one load (200,000 psi) using both unembrittled and embrittled specimens and varying the rate of cycling between 0.33 and 2500 rpm. A few additional values were also available from previous tests for 250 and 10,000 rpm respectively.



| Specimen No. | Strength | Stress |
|---|---|---|
| Unemb. 22 | 250 000 psi | 175 000 psi |
| Embr. 24 | 250 000 psi | 175 000 psi |
| Unemb. 37 | 21 0 000 psi | 175 000 psi |
| Embr. 34 | 21 0 000 psi | 175 000 psi |

FIG. 6.—Deflection of Notched Unembrittled and Embrittled Specimens Heat Treated to 250,000 and 210,000 psi Strength Level *versus* Number of Cycles.

The results of these tests are assembled in Fig. 5. This graph discloses the following:

1. The rate effect is rather small for unembrittled specimens. It is practically nil up to about 1000 rpm, while at still higher cycling rates the number of cycles to failure appears to be slightly greater, perhaps as much as 75 per cent.

2. A decrease in the number of cycles to failure due to hydrogen is generally pronounced at low cycling rates, while it fades out completely at high cycling rates.

3. It appears, from the tests performed so far, see Fig. 5, that the scattering of data obtained from specimens which were embrittled and tested, under practically identical conditions, within a relatively short time period was much smaller than that observed during tests extended over a longer time period. No attempt has yet been made to establish the factors responsible for this inconsistency.

### *The Bending Deflection of Notched Specimens During Cycling:*

In order to obtain some information on the mechanism of fatigue failure in hydrogen-embrittled specimens, a variety of further studies was undertaken.

In a number of instances, the deflection during cycles was recorded. In the case of smooth specimens, generally first a certain increase in deflection was observed, which then became rather small for a major portion of the specimen life, and which finally led to a rapid acceleration of the deflection shortly before failure. Unembrittled specimens usually exhibited a more extended final period than embrittled specimens. Otherwise, the evaluation of about 35 specimens did not disclose anything significant.

The deflection of notched specimens, on the other hand, was found to increase at a nearly constant rate for at least 50 per cent of the specimen life, as illustrated for a few examples in Fig. 6. A very short period of rapidly increasing deflection preceded this constant-rate change, and a somewhat more extended period of accelerated deflection indicated the subsequent failure. In Fig. 7, the rate of increase in deflection per cycle is plotted *versus* the applied stress, with the strength level being the parameter. The graph reveals that the rate effect increases progressively, as the applied stress becomes greater. As mentioned above, the 250,000-psi strength level represents the steel condition most sensitive to hydrogen embrittlement. In Fig. 8, the constant increase in deflection per cycle is plotted *versus* strength level for two loads (175,000 and 225,000 psi) and for both embrittled and unembrittled specimens. The following can be deduced from this graph:

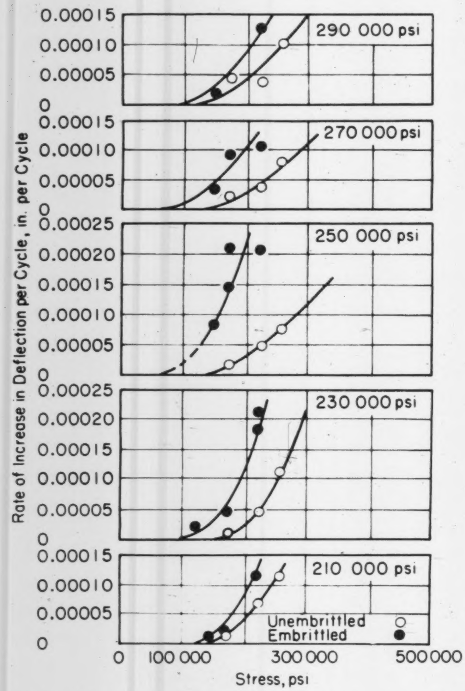


FIG. 7.—Rate of Increase in Deflection per Cycle *versus* Stress for 4340 Steel Heat Treated to Strength Levels Between 290,000 and 210,000 psi.

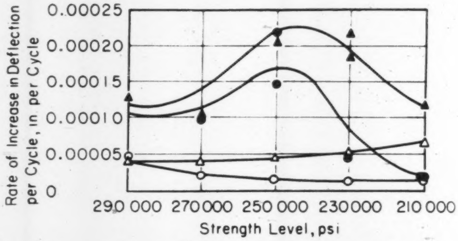


FIG. 8.—Rate of Increase in Deflection per Cycle of Notched Embrittled and Unembrittled Specimens *versus* Strength Level.

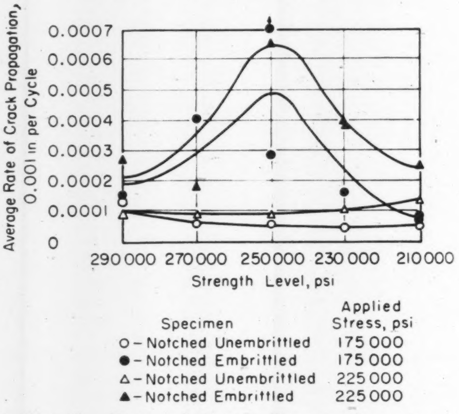


FIG. 9.—Rate of Crack Propagation *versus* Strength Level.

1. The deflection of unembrittled (notched) specimens increases during cycling at a rate that decreases for the smaller applied stress but increases for the larger stress as the strength level decreases.

2. The presence of hydrogen causes the deflection to increase at a higher rate, other conditions being equal.

3. This effect of hydrogen is greatest for the steel tempered to 250,000 psi, while it is rather small if the steel is heat treated to a strength level of 210,000 psi.
4. The relative effect of hydrogen is (slightly) more pronounced at a stress of 225,000 psi than at a stress of 175,000 psi.

The primary reason for the gradual changes in deflection observed and discussed above, is the progressing development of the fatigue failure. In smooth steel specimens, a fatigue crack is usually observed only after the cycling approaches failure; and it then progresses at a rapidly increasing rate. In contrast, the fatigue failure of the notched steel specimens could be readily detected after a small fraction of the cycle numbers to failure, and it grew toward the center at a certain rate. This explains the similarity of the curves in Fig. 8 to those in Fig. 9, which illustrates the average rate of fatigue-crack propagation, and which is discussed later in more detail.

*Crack Propagation:*

In order to obtain quantitative information on crack development, a few test specimens were subjected to different numbers of cycles at a constant load. Notched specimens were selected for this study, as surface cracks in these can be detected after a limited number of cycles. In contrast, surface cracks in unbroken smooth specimens have not been observed in these experiments. Four unembrittled and two embrittled fatigue specimens of the 4340 steel, heat treated to 230,000 psi, and run for various numbers of cycles at a stress of 225,000 psi were used for this analysis. The crack depth was determined by longitudinally sectioning the test specimens along two planes, under 90 deg to each other, taking photomicrographs of the four

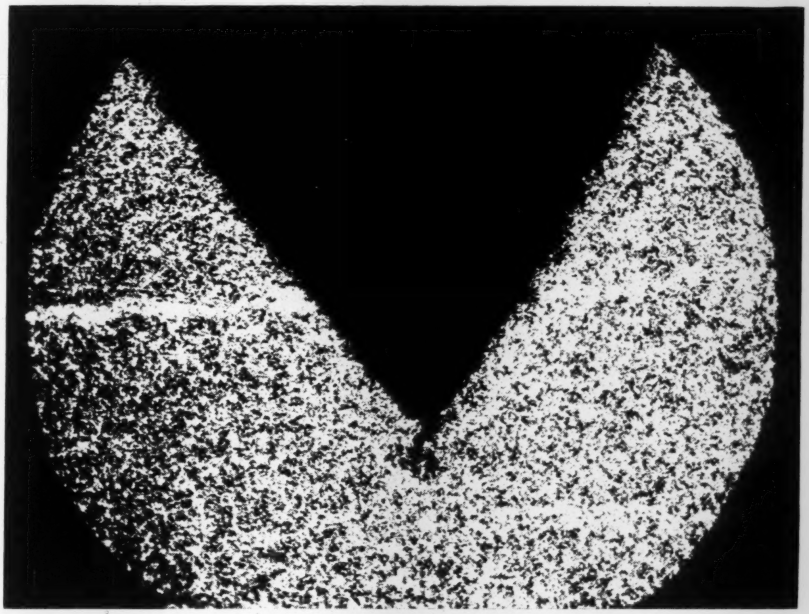


FIG. 10.—Longitudinal Section of Specimen X-28 (× 100). Unembrittled, 230,000 psi strength 52 cycles at 225,000 psi applied stress.

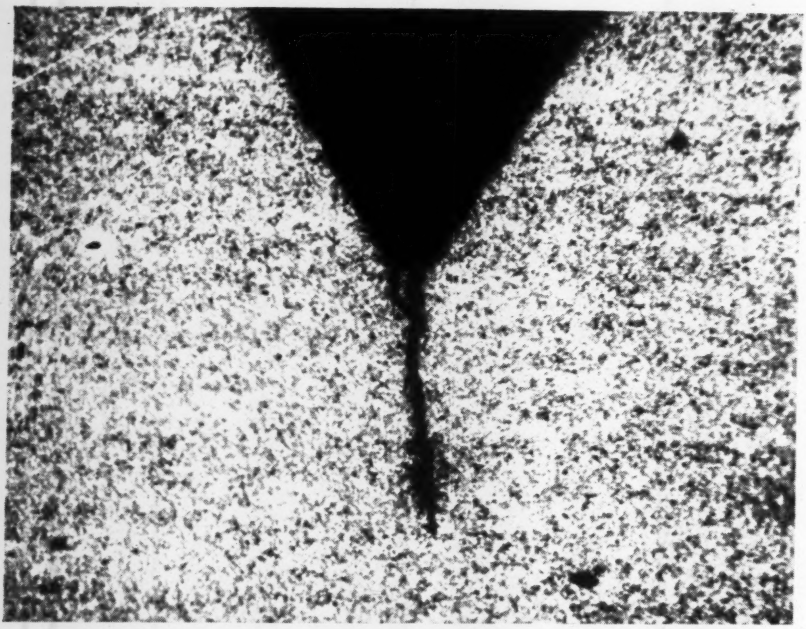


FIG. 11.—Longitudinal Section of Specimen X-26 (× 100). Unembrittled, 230,000 psi strength 190 cycles at 225,000 psi applied stress.

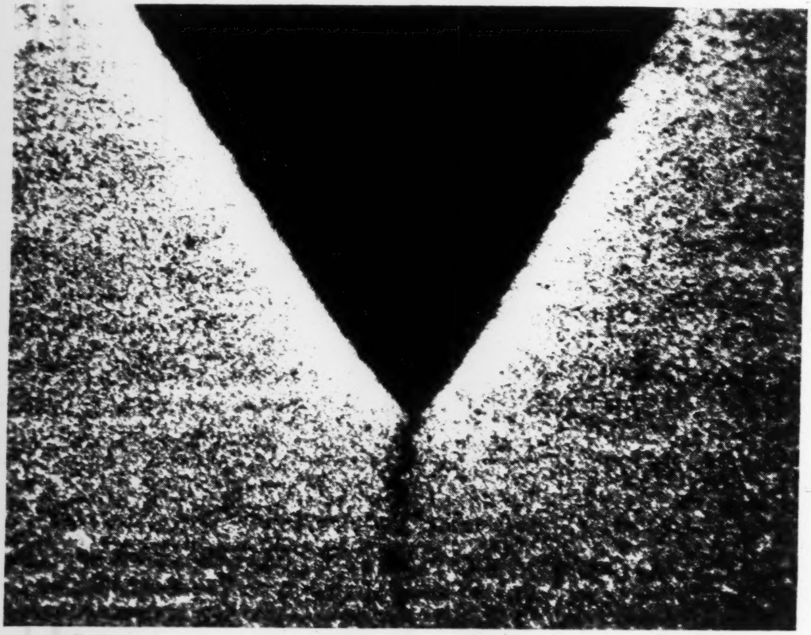


FIG. 12.—Longitudinal Section of Specimen X-50 (× 100). Embrittled, 230,000 psi strength, 50 cycles at 225,000 psi applied stress.

crack areas thus exposed, and measuring the crack depth.

Figures 10 to 12 illustrate the crack in several instances. The average values of crack depth are plotted in Fig. 13 as a function of the cycle number. Added is the crack depth of broken specimens which was determined from macrographs, as further discussed below.

This graph clearly reveals the following:

1. The crack appears to be initiated after very few cycles. It then progresses in the radial direction practically at a constant linear rate until at least one half of the specimen life is expended. Finally, the crack penetration becomes increasingly faster, until the rest of the section suddenly fractures, probably within less than one cycle, that is statically.

2. Hydrogen causes, for the conditions of material and load used in these tests, a considerable acceleration of crack propagation. The cracking rate of the hydrogen-loaded material is found to be about three times as high as that of the unembrittled material.

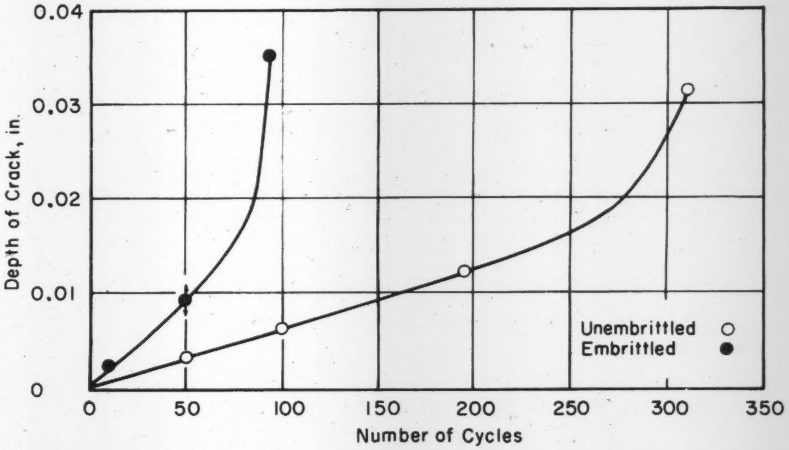


FIG. 13.—Depth of Crack *versus* Number of Cycles for Unembrittled and Embrittled Notched Rotating Beam Specimen Tempered to a Strength Level of 230,000 psi (700 F Tempering Temperature) Tested at a Cycling Stress of 225,000 psi.

In order to evaluate these effects further, a "mean crack propagation rate" is introduced, being the ratio of the (radial) crack length to the number of cycles to failure. This rate is, according to Fig. 10, approximaetly 1.5 times the actual rate of crack propagation in the beginning.

The representation of this mean crack propagation rate *versus* strength level in Fig. 9 results in much the same relations as already discussed for the increase in bending deflection, see Fig. 8. This close correlation is further illustrated by Fig. 14, which indicates that the rates of increase in bending deflection and of crack

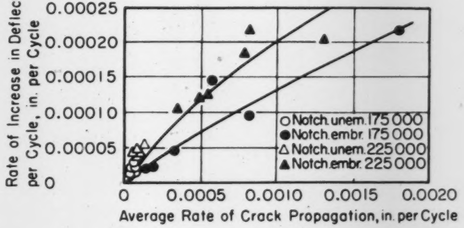
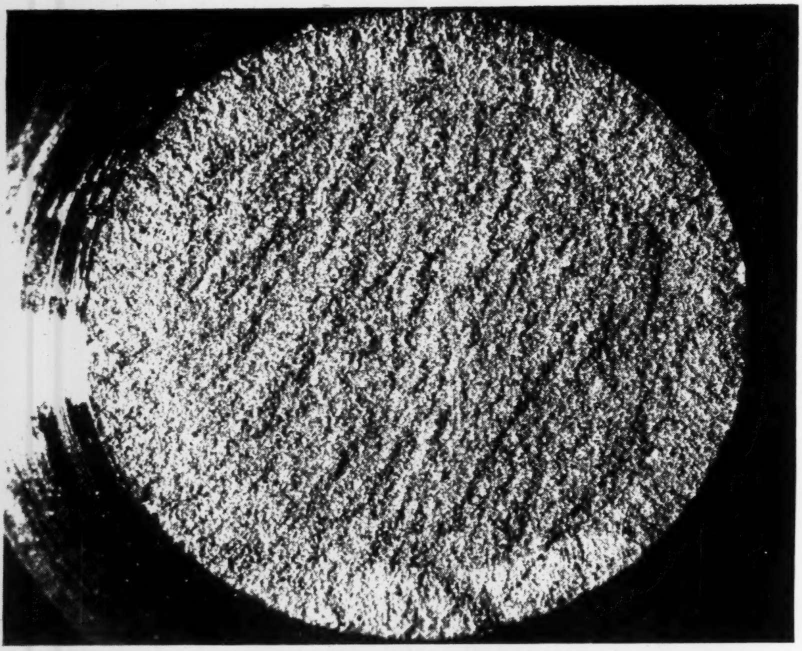
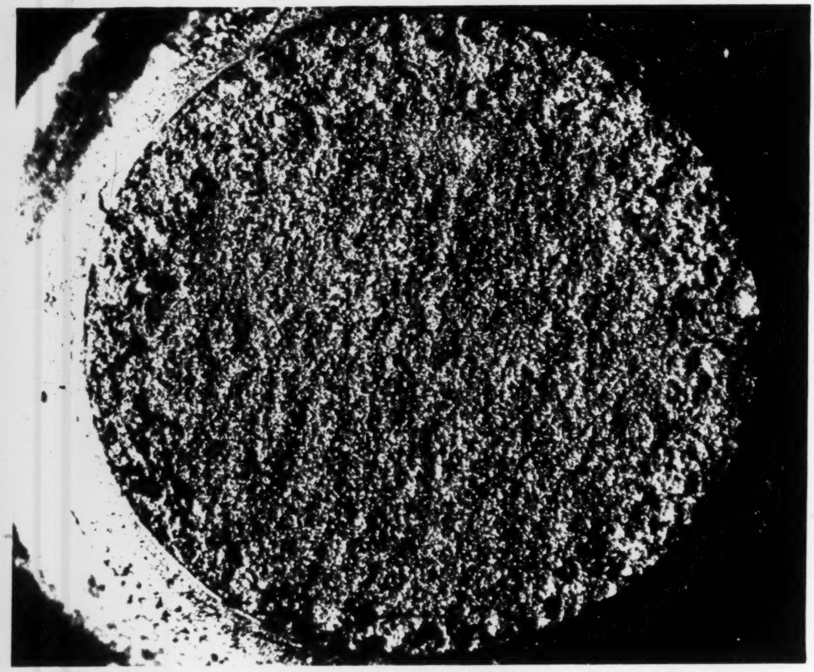


FIG. 14.—Rate of Increase in Deflection per Cycle *versus* Average Rate of Crack Propagation.

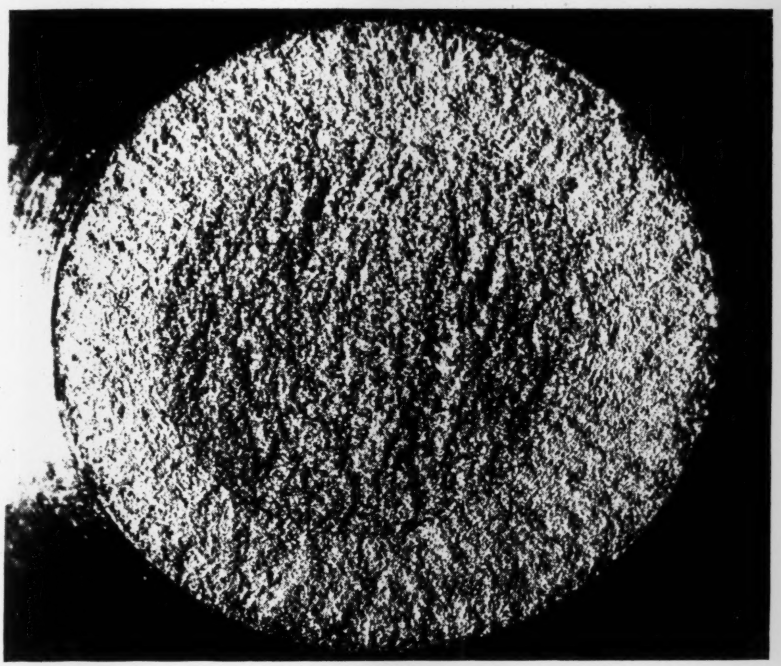


Specimen No. 6, unembrittled, 314 cycles

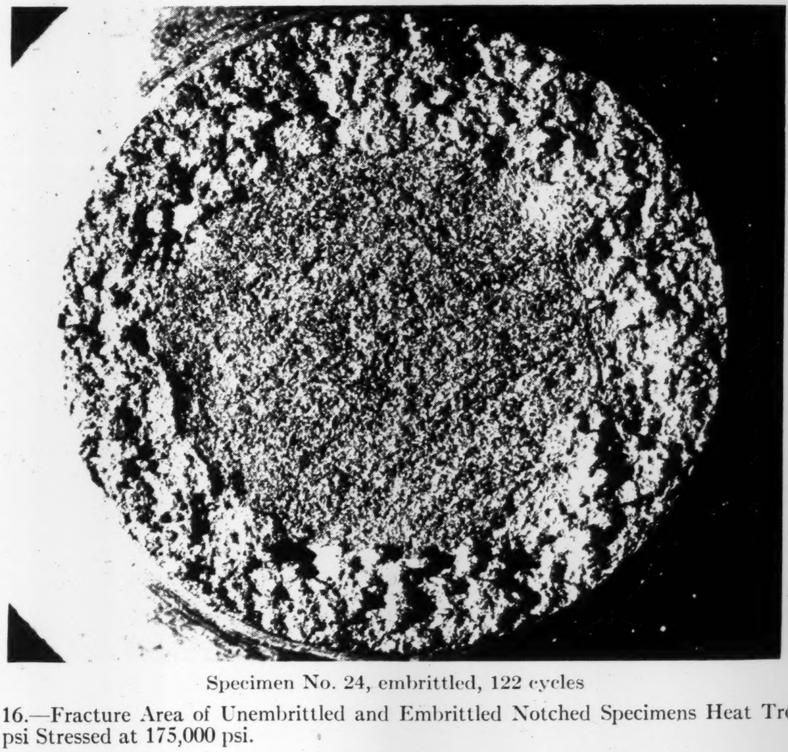


Specimen No 3, embrittled, 114 cycles

FIG. 15.—Fracture Area of Unembrittled and Embrittled Notched Specimens Heat Treated to 290,000 psi Stressed at 175,000 psi.

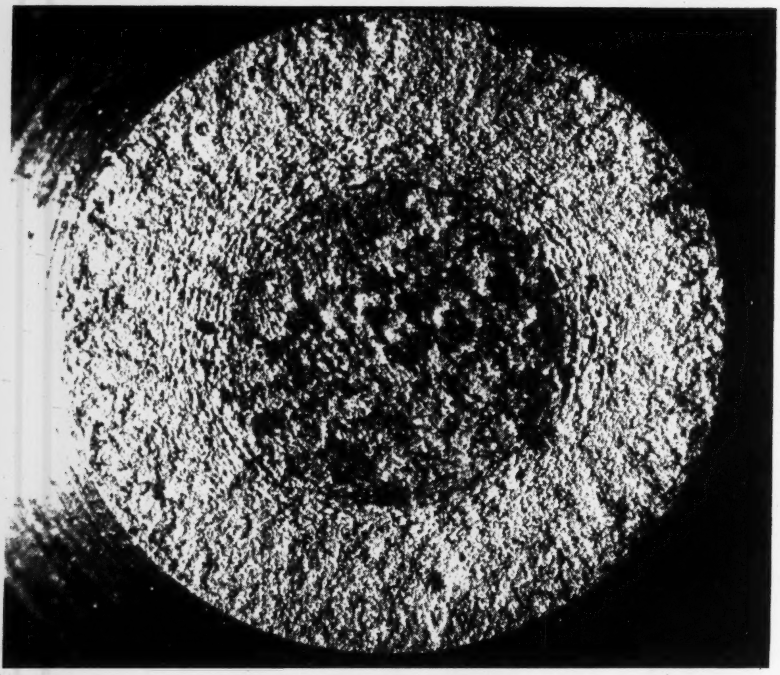


Specimen No. 22, embrittled, 122 cycles

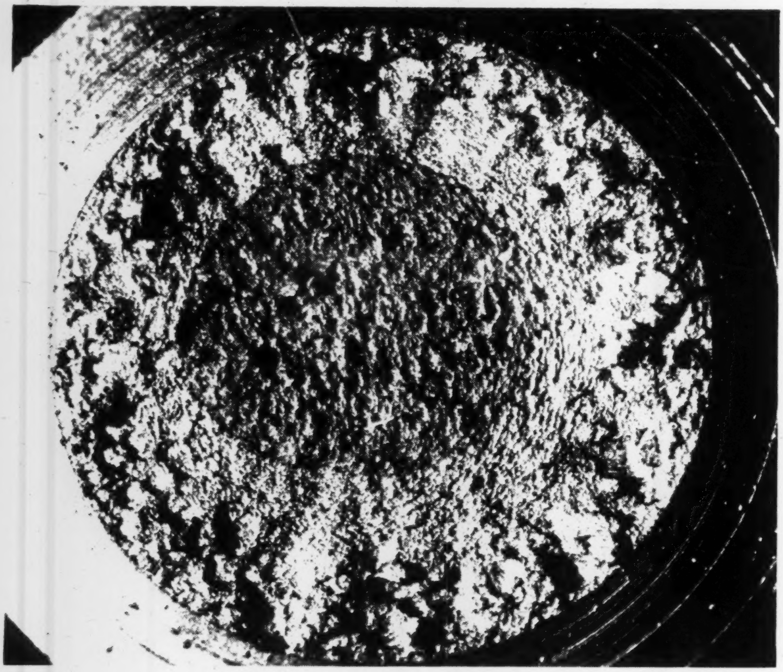


Specimen No. 24, embrittled, 122 cycles

FIG. 16.—Fracture Area of Unembrittled and Embrittled Notched Specimens Heat Treated to 250,000 psi Stressed at 175,000 psi.



Specimen No. 37, unembrittled, 930 cycles



Specimen No. 39, embrittled, 611 cycles

FIG. 17.—Fracture Area of Unembrittled and Embrittled Notched Specimens Heat Treated to 210,000 psi Stressed at 175,000 psi.

propagation are nearly proportional to each other, at any given load, and independent of the strength level.

Because of the extensive amount of work required for the quantitative determination of crack propagation under the various conditions investigated, an attempt was made to arrive at some such information simply from the appearance of the failed specimens.

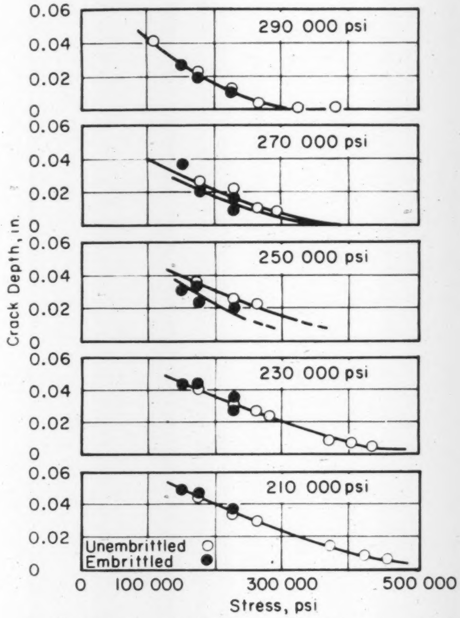


Fig. 18.—Crack Depth *versus* Stress for 4340 Steel Heat Treated to Strength Levels Between 290,000 and 210,000 psi at the Moment of Failure.

A number of these failures of notched specimens, are illustrated in Figs. 15 to 17. These reveal the generally known fact that the fatigue crack progresses only to a certain depth, while the remainder comprises a static failure, as the strength of the section which is weakened by the fatigue crack becomes unable to maintain the applied bending load. In addition, these photographs show the following:

1. The appearance of the fatigue crack in embrittled specimens is rather different from those in unembrittled material. In the latter case, the annular fatigued area looks much the same as the statically fractured core, except that faint concentric marks are visible in the annulus. In contrast, the fatigue crack of the hydrogen-containing specimens is always observed to be rather rough, consisting of comparatively deep valleys and high peaks. The true nature of this fracture structure has not yet been determined. The core, however, is very similar in both instances.

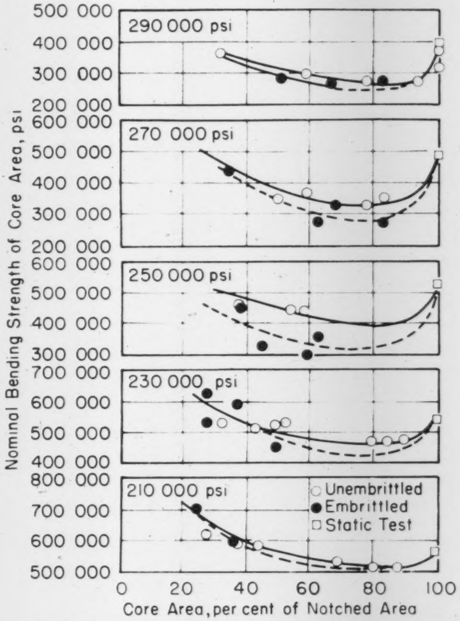


Fig. 19.—Nominal Bending Strength of Core Area *versus* Core Area for 4340 Steel Heat Treated to Strength Levels Between 290,000 and 210,000 psi.

2. The presence of hydrogen also causes the crack to develop to a certain depth faster than in unembrittled material.

*Crack Depth and Static Strength:*

Another problem in fatigue relates to the cross-section of the final core and its strength. By multiplying the applied stress with the ratio of initial to final cross-section, a nominal static bend strength, at the moment of failure is obtained. These data are obtained from Fig. 18, which presents the crack depth (in inches) as a function of the applied stress.

The values of nominal bend strength are plotted in Fig. 19 *versus* the cross-sectional area of the static failure. The curves for the different strength levels are rather similar. The introduction of hydrogen only slightly reduces this "core strength" and distinctly only for the two strength levels of 250,000 and 270,000 psi.

To further evaluate the problem of core strength, the (nominal) bend strength has been divided by a value equal to twice the tensile strength. The latter value is approximately equal to the bend strength of a smooth and ductile specimen (6). This ratio of core strength to twice the tensile strength, therefore, has the significance of a "notch strength ratio". Its values are plotted in Fig. 20 *versus* the percentage of crack area ("Notch depth"). These curves are indeed, rather similar to those of the notch strength ratio *versus* notch depth, previously obtained for heat treated steels. However, it must be considered that the origin of each curve is the notch strength ratio of a specimen provided with a sharp machined notch having a depth of 50 per cent. The graph shows that the development of a shallow crack first gradually reduces the nominal bend strength. This is in agreement with the concept that a crack represents a higher stress concentration than the machined notch, but that this difference only gradually appears as the notch increases in depth. Subsequently, after having reached a certain depth, the nominal bend strength progressively increases. This, again, is in agreement with the previously established fact that the strength of deeply-notched specimens increases with increasing notch depth, presumably because of the effect of increasing triaxiality.

Regarding the effect of tempering temperature, Fig. 20 illustrates that the notch strength ratio in bending increases with decreasing strength level, and at any given crack depth, including zero, at approximately the same rate. This can also be expressed by the statement that the nominal bend strength of a cracked, and, during the fatigue test, broken specimen depends upon the notch ductility of the metal which had been determined by a notch tension test.

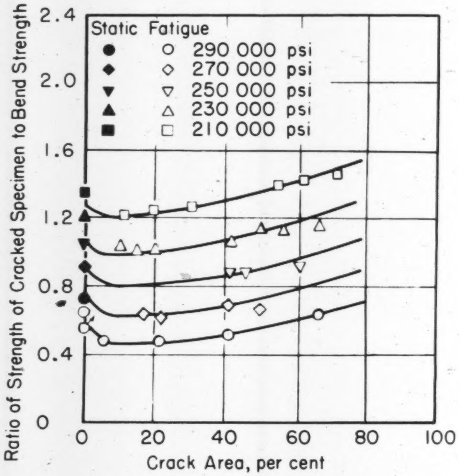


FIG. 20.—Notch Strength Ratio in Bending *versus* Crack Area for 4340 Steel Heat Treated to Strength Levels Between 290,000 and 210,000 psi.

A deficiency of these results and conclusions is the fact that they are based on cross-sections which may derive additional strength from the mutual contact of the crack surface at the compressed portion of the section. However, this does not affect the pualitative conclusions discussed above.

## DISCUSSION AND CONCLUSIONS

The investigation revealed the following relations regarding the decrease in fatigue strength, for a given number of cycles, or in the number of cycles for a given applied stress, caused by the introduction of hydrogen in plating.

In regard to all different variables, it is noted that these hydrogen embrittlement phenomena are limited to a certain range in the value of each variable. Whenever an effect was observed, the following applied:

1. The hydrogen embrittlement effect was larger for notched than for unnotched (smooth) specimens.
2. SAE 4340 Steel is most susceptible to hydrogen-embrittlement, if heat-treated to approximately 250,000 ± 20,000 psi tensile strength.
3. The hydrogen influence increases as the frequency of stress applications decreases.
4. The hydrogen-embrittlement effect increases as the applied stress increases.

Regarding the effect of hydrogen-embrittlement on the mechanism of crack propagation and fracture, the following observations were made:

1. The presence of hydrogen generally accelerates the crack propagation during the fatigue life of the specimen.
2. The fatigue break of a hydrogen-embrittled specimen has a much rougher appearance than that of an unembrittled specimen. This applies also in instances where the reduction in fatigue life due to hydrogen is small. In contrast, the final static fracture of both hydrogen-embrittled and unembrittled specimens appears approximately the same.
3. The static strength of the core of a partly cracked specimen depends upon its cross-sectional area in much the same manner as the strength of an intentionally notched specimen.

*Acknowledgment:*

The authors are indebted to R. Bale, for assistance in the experimental work, and to B. B. Muvdi, for assistance in the selection of materials and procedures.

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## REFERENCES

(1) G. Sachs and W. Beck, "Survey of Low Alloy Aircraft Steels Heat Treated to High-Strength Levels, Part 1: Hydrogen Embrittlement," Wright Air Development Center, *Technical Report 53-254*, Part 1 (1953).

(2) W. Beck, E. P. Klier, and G. Sachs, "Constant Strain Rate Bend Tests on Hydrogen-Embrittled High Strength Steels," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., *Journal of Metals*, Oct., 1956, p. 1263.

(3) B. B. Muvdi, E. P. Klier, and G. Sachs, "Design Properties of High Strength Steel in the Presence of Stress Concentrations and Hydrogen Embrittlement, Part 1: Effects of Hydrogen Embrittlement on High Strength Steels—Static Properties," Wright Air Development Center, *Technical Report 55-18*, Part 1 (1954).

(4) B. B. Muvdi, E. P. Klier, and G. Sachs, "Design Properties of High Strength Steel in the Presence of Stress Concentrations," Wright Air Development Center, *Technical Report 55-103* (1955).

(5) B. B. Muvdi, E. P. Klier, and G. Sachs, "Design Properties of High Strength Steels in the Presence of Stress Concentrations, Part 2: Axial Load Fatigue Properties of High Strength Steels," Wright Air Development Center, *Technical Report 56-395*, Part 2 (1956).

(6) G. Sachs and G. Scheven, "Relation Between Direct-Stress and Bending Fatigue of High-Strength Steels," see p. 667 this publication.

# SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON LARGE FATIGUE TESTING MACHINES AND THEIR RESULTS

The papers and discussions in this Symposium were presented at the Eighth and Tenth Sessions of the Sixtieth Annual Meeting of the American Society for Testing Materials, held in Atlantic City, N. J., June 18, 1957. It was sponsored by Committee E-9 on Fatigue, with D. G. Richards and O. J. Horger presiding over the Eighth Session and J. F. Millan and H. J. Grover presiding over the Tenth Session. The symposium chairman was J. M. Lessells.

The papers included in the Symposium are:

Introduction—J. M. Lessells
A Unique Machine for Large Scale Fatigue Testing—H. V. Cordiano
Torsion Fatigue Testing of Axle Shafts—E. J. Eckert
Fatigue Testing of Airframe Structural Components—H. W. Foster
Fatigue Performance of Marine Shafting—Laboratory and Service Tests—T. W. Bunyan
Fretting Corrosion of Large Shafts as Influenced by Surface Treatments—O. J. Horger and H. R. Neifert
Fatigue Tests of Large Alloy Steel Shafts—F. C. Eaton
Influence of Operating Experience and Full Scale Tests on Propulsion Shafting Design of U. S. Navy Ships—R. Michel
On Reasons of Sudden Fracture of Machine Parts and Structure Elements—G. V. Uzhik, M. J. Galperin, and A. A. Zooykova
On Determination of Dynamical Loading in Full Size Fatigue Tests and Some Results—S. V. Serensen and M. E. Garf

These papers with discussion were issued as *ASTM Special Technical Publication No. 216*, entitled "Symposium on Large Fatigue Testing Machines and Their Results."

# EFFECT OF PHOSPHORUS ON THE SUSCEPTIBILITY TO TEMPER EMBRITTLEMENT OF CAST Cr-Mo-V STEEL*

By J. Chaberek[1] and R. S. Zeno[1]

## Synopsis

This investigation was conducted to determine the effect of phosphorus content on the toughness and susceptibility to temper embrittlement of cast 1% Cr-1% Mo-$\frac{1}{4}$% V basic electric steel. Test material at each phosphorus level (0.008, 0.02, 0.05, and 0.08 nominal weight per cent) was isothermally aged at 900 F and 950 F for 1000 hr. V-notch Charpy impact transition temperatures (used as a basis for determining susceptibility to temper embrittlement) were obtained for each phosphorus level and embrittling heat treatment. Room temperature tensile properties were determined for all material isothermally aged. It was found that isothermally aged material at phosphorus levels higher than 0.02 per cent were susceptible to temper embrittlement.

A study of the effect of phosphorus content on the susceptibility to temper embrittlement of low-alloy steel is essential if optimum mechanical properties are to be developed in heavy section castings. In general, the role of phosphorus on the susceptibility to temper brittleness of these steels appears to be a complex one. Preece and Carter (**1**)[2] investigated the effect of two levels of phosphorus (0.001 and 0.036 per cent) on the temper brittleness of a high-purity iron. Material at both phosphorus levels was isothermally aged at 500 C (932 F) for 144 hr. No transition temperature rise was shown in either of these materials with or without the embrittling heat treatment. However, they showed that with 1.5 per cent chromium added and with phosphorus increased from 0.001 to 0.048 per cent the transition temperature increased 40 C in the unembrittled and 115 C in the embrittled state. Also, they reported that the addition of molybdenum decreased the susceptibility to temper brittleness.

This investigation was conducted to determine the effect of different levels of phosphorus content on the toughness and susceptibility to temper embrittlement of cast chromium-molybdenum-vanadium (Cr-Mo-V) steel. The effect of various phosphorus contents on the room temperature tensile properties was also investigated.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

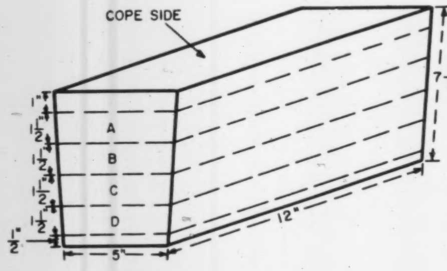
[1] Materials and Processes Laboratory, Large Steam Turbine-Generator Department, General Electric Co., Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 713.

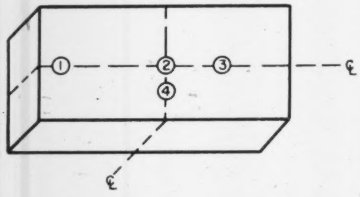
## PROCEDURE

### *Material:*

A base heat of silicon deoxidized Cr-Mo-V steel, produced by the basic electric process, was used in this investigation. Eight ingots, each weighing approximately 135 lb were poured from heat 2733. Varying amounts of phosphorus (in the form of iron phosphide) were added to each mold during the pouring. Immediately after each phosphorus addition the molten metal was stirred with a steel rod to assure thorough mixing and uniformity of composition.



SECTIONING OF TEST INGOTS



LOCATION OF SAMPLES REMOVED FROM TOP SURFACE OF SLABS A, B, C AND D

FIG. 1.— Sectioning and Sampling of Test Ingots—Heat 2733.

The following is the chemical composition of heat 2733 in weight per cent:

| Carbon | Silicon | Manganese | Sulfur | Phosphorus | Chromium | Nickel | Molybdenum | Vanadium |
|---|---|---|---|---|---|---|---|---|
| 0.17 | 0.42 | 0.62 | 0.008 | 0.008 | 1.04 | 0.10 | 1.04 | 0.19 |

### *Sectioning and Phosphorus Analyses:*

Each ingot was sectioned into four slabs marked A, B, C, and D. As phosphorus is one of the elements most likely to segregate in steel ingots, four samples were removed from each slab for phosphorus determinations as shown in Fig. 1. Test material was selected on the basis of these determinations (Table I). Sample numbers were determined by the ingot, slab, and position from which each sample was removed; therefore, 2A-1 is the sample from ingot 2, slab A, position 1.

### *Heat Treatment:*

Before sectioning, the eight ingots were austenitized at 1050 C (1922 F) for 12 hr and air cooled. After sectioning, the test material at each phosphorus level (0.008, 0.02, 0.05, and 0.08 nominal weight per cent) was given the following heat treatment:

1. Temper at 690 C (1275 F) and oil quench.
2. Temper at 690 C (1275 F), oil quench, and isothermally age at 482 C (900 F) for 1000 hr.
3. Temper at 690 C (1275 F), oil quench, and isothermally age at 510 C (950 F) for 1000 hr.

Material at the 0.008 per cent phosphorus level was tempered at 690 C (1275 F) and furnace cooled at 20 C per hr.

### *Testing:*

Cr-Mo-V steels at various levels of phosphorus were compared on the basis of the results obtained from the following tests:

(*a*) V-notch Charpy (transition curves).

(*b*) Susceptibility to temper embrittlement (measured by the increase in fracture-appearance transition temperature).

(*c*) Room temperature tension.

(*d*) Metallographic examination.

## RESULTS AND DISCUSSION

In this study, the degree of temper brittleness is assumed to be proportional to the rise in the fracture-appearance transition temperature of an embrittled material above the transition temperature of an unembrittled standard. Specimens oil quenched from the tempering temperature provided an unembrittled standard. The fracture-appearance transition temperature was based on 50 per cent fibrous fracture.

TABLE I.—PHOSPHORUS ANALYSES OF CAST Cr-Mo-V EXPERIMENTAL INGOTS.

| Sample | Weight, per cent | Sample | Weight, per cent | Sample | Weight, per cent |
|---|---|---|---|---|---|
| 2 A-1 | 0.007 | 10 C-1 | 0.020 | 5 C-1 | 0.048 |
| 2 A-2 | 0.010 | 10 C-2 | 0.017 | 5 C-2 | 0.052 |
| 2 A-3 | 0.009 | 10 C-3 | 0.016 | 5 C-3 | 0.048 |
| 2 A-4 | 0.007 | 10 C-4 | 0.019 | 5 C-4 | 0.050 |
| 2 B-1 | 0.008 | 10 D-1 | 0.022 | 7 A-1 | 0.044 |
| 2 B-2 | 0.013 | 10 D-2 | 0.022 | 7 A-2 | 0.049 |
| 2 B-3 | 0.008 | 10 D-3 | 0.019 | 7 A-3 | 0.051 |
| 2 B-4 | 0.005 | 10 D-4 | 0.021 | 7 A-4 | 0.051 |
| 2 C-1 | 0.008 | 4 C-1 | 0.023 | 6 A-1 | 0.089 |
| 2 C-2 | 0.006 | 4 C-2 | 0.017 | 6 A-2 | 0.078 |
| 2 C-3 | 0.006 | 4 C-3 | 0.023 | 6 A-3 | 0.088 |
| 2 C-4 | 0.006 | 4 C-4 | 0.022 | 6 A-4 | 0.082 |
| 2 D-1 | 0.007 | 5 A-1 | 0.055 | 6 C-1 | 0.073 |
| 2 D-2 | 0.006 | 5 A-2 | 0.047 | 6 C-2 | 0.067 |
| 2 D-3 | 0.006 | 5 A-3 | 0.048 | 6 C-3 | 0.079 |
| 2 D-4 | 0.008 | 5 A-4 | 0.057 | 6 C-4 | 0.080 |
| 10 B-1 | 0.018 | 5 B-1 | 0.047 | | |
| 10 B-2 | 0.016 | 5 B-2 | 0.055 | | |
| 10 B-3 | 0.017 | 5 B-3 | 0.055 | | |
| 10 B-4 | 0.016 | 5 B-4 | 0.047 | | |

Since susceptibility to temper embrittlement is affected by hardness and austenitic grain size, in the work reported here, an attempt was made to keep these properties uniform at each phosphorus level. The austenitic ASTM grain size ranged from 3 to 4. The Brinell hardnesses were in the range of 225 to 233 with the exception of the 0.08 per cent phosphorus material which was 207.

*Unaged Material:*

Jolivet and Vidal (2) showed that for a 0.25 per cent carbon, 0.3 manganese, 1.4 chromium steel an increase in the phosphorus content from 0.012 to 0.044 per cent resulted in an impact transition temperature rise of 50 C (90 F). Hultgren and Chang (3) reported that an increase in phosphorus from 0.012 to 0.065 per cent in a 0.17 carbon, 3 chromium steel resulted in an impact transition temperature rise of 105 C (189 F).

The V-notch Charpy results of increasing phosphorus from 0.008 to 0.08 per cent in an unaged Cr-Mo-V steel are presented in Table II and Figs. 2, 3, and 4. The fracture-appearance transition temperatures are summarized in Table III. These data indicate that increasing phosphorus does not significantly increase the transition temperature of the unaged steel.

Powers (4) has indicated that in a 0.25 per cent carbon, 1 manganese, 1 chromium steel the addition of 0.5 and 1.0 per cent molybdenum lowered the transition temperature 78 C (140 F) and 75 C (135 F), respectively. Baeyertz, Craig, and Sheehan (5) investigated the effect of phosphorus and molybdenum on the toughness of a 5140 steel. They found that increasing phosphorus from 0.020 to 0.035 per cent resulted in a loss of toughness. However, for a given hardenability this loss in toughness was partially compensated by replacing part of the chromium with molybdenum. From these results it appears that molybdenum counteracts the detrimental effects of increasing phosphorus. Our results are in agreement with these investigators in that no significant transition temperature rise occurred with an increase in phosphorus in an unaged Cr-Mo-V steel.

TABLE II.—V-NOTCH CHARPY RESULTS ON UNAGED AND AGED CAST Cr-Mo-V STEELS.

| Phosphorus Content, nominal weight per cent | Brinell Hardness Number[a] | ASTM Austenitic Grain Size | Aging Temperature, deg Cent (deg Fahr) | | 25 C (77 F) | 75 C (167 F) | 100 C (212 F) | 150 C (302 F) | 200 C (392 F) | 250 C (482 F) |
|---|---|---|---|---|---|---|---|---|---|---|
| 0.01...... | 228 | 3 to 4 | Unaged | Energy, ft-lb............... | 6 | 29 | 44 | 89 | 88 | |
| | | | | Per cent fibrous fracture...... | 0 | 10 | 20 | 93 | 100 | |
| 0.01...... | | | 482 (900) | Energy, ft-lb............... | | 23 | 27 | 74 | | 97 |
| | | | | Per cent fibrous fracture...... | | 5 | 20 | 80 | | 100 |
| 0.01...... | | | 510 (950) | Energy, ft-lb............... | | 15 | 31 | 74 | | 80 |
| | | | | Per cent fibrous fracture...... | | 5 | 15 | 80 | | 100 |
| 0.02...... | 233 | 3 to 4 | Unaged | Energy, ft-lb............... | 11 | 33 | 42 | 98 | | |
| | | | | Per cent fibrous fracture...... | 0 | 10 | 30 | 100 | | |
| 0.02...... | | | 482 (900) | Energy, ft-lb............... | | 15 | 39 | 65 | | 77 |
| | | | | Per cent fibrous fracture...... | | 5 | 15 | 80 | | 100 |
| 0.02...... | | | 510 (950) | Energy, ft-lb............... | | 16 | 34 | 62 | | 83 |
| | | | | Per cent fibrous fracture...... | | 5 | 15 | 80 | | 100 |
| 0.05...... | 225 | 3 to 4 | Unaged | Energy, ft-lb............... | 6 | 26 | 55 | 62 | 85 | |
| | | | | Per cent fibrous fracture...... | 0 | 6 | 35 | 75 | 100 | |
| 0.05...... | | | 482 (900) | Energy, ft-lb............... | | 12 | 16 | 40 | | 68 |
| | | | | Per cent fibrous fracture...... | | 3 | 8 | 40 | | 100 |
| 0.05...... | | | 510 (950) | Energy, ft-lb............... | | 12 | 13 | 38 | | 84 |
| | | | | Per cent fibrous fracture...... | | 5 | 8 | 50 | | 100 |
| 0.08...... | 207 | 3 to 4 | Unaged | Energy, ft-lb............... | 12 | 44 | 60 | 95 | | |
| | | | | Per cent fibrous fracture...... | 2 | 15 | 40 | 100 | | |
| 0.08...... | | | 482 (900) | Energy, ft-lb............... | | 7 | 11 | 40 | | 65 |
| | | | | Per cent fibrous fracture...... | | 2 | 8 | 50 | | 100 |
| 0.08...... | | | 510 (950) | Energy, ft-lb............... | | 17 | 42 | 76 | | 98 |
| | | | | Per cent fibrous fracture...... | | 5 | 20 | 70 | | 100 |

[a] Hardness of Charpy bars (average of 3 readings).

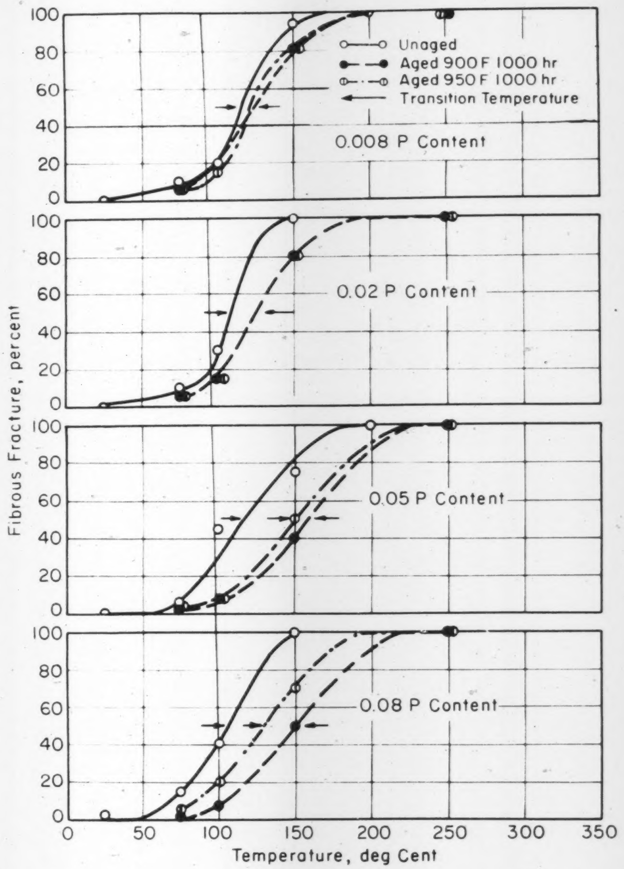


FIG. 2.—Effect of Phosphorus on the Fracture Appearance Transition Temperature of a Cast Cr-Mo-V Steel.

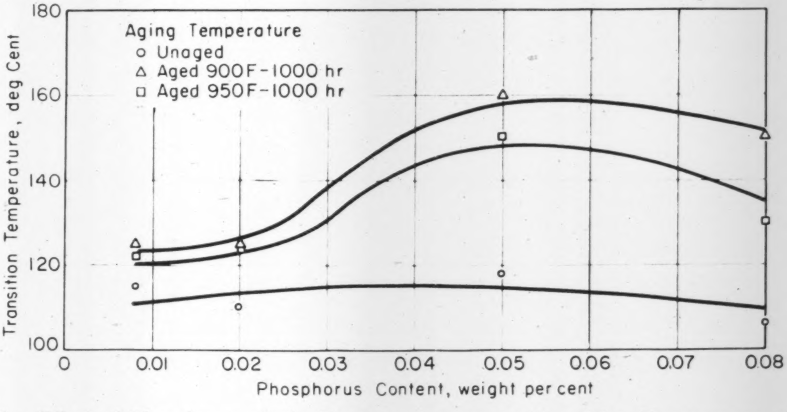


FIG. 3.—Effect of Phosphorus on the Fracture Appearance Transition Temperature of a Cast Cr-Mo-V Steel.

*Isothermally Aged Material:*

V-notch Charpy data on the aged material are summarized in Table II, and the fracture-appearance transition curves are shown in Fig. 2. The fracture-appearance transition temperatures of the aged material are summarized in Table III and presented in Fig. 3.

At the 0.008 and 0.02 per cent phosphorus levels, materials aged isothermally at 482 C (900 F) and 510 C (950 F) for 1000 hr showed no significant rise in the transition temperature. At the higher phosphorus levels (0.05 and 0.08 per cent) a marked rise in transition temperature occurred during both aging heat treatments.

The lower transition temperatures of the 0.08 per cent phosphorus materials in the aged condition were due in part to the lower hardness level (Fig. 3 and Table II). Slightly more embrittlement occurred during aging at 482 C (900 F) than at 510 C (950 F), Figs. 2 and 3. Material at the 0.08 per cent phosphorus level, aged at 482 C (900 F), shows a substantial decrease in the impact strength (Fig. 4 and Table II). Other investigators (1, 2, 3, 5, 6, 7) have shown that an increase in the phosphorus con-

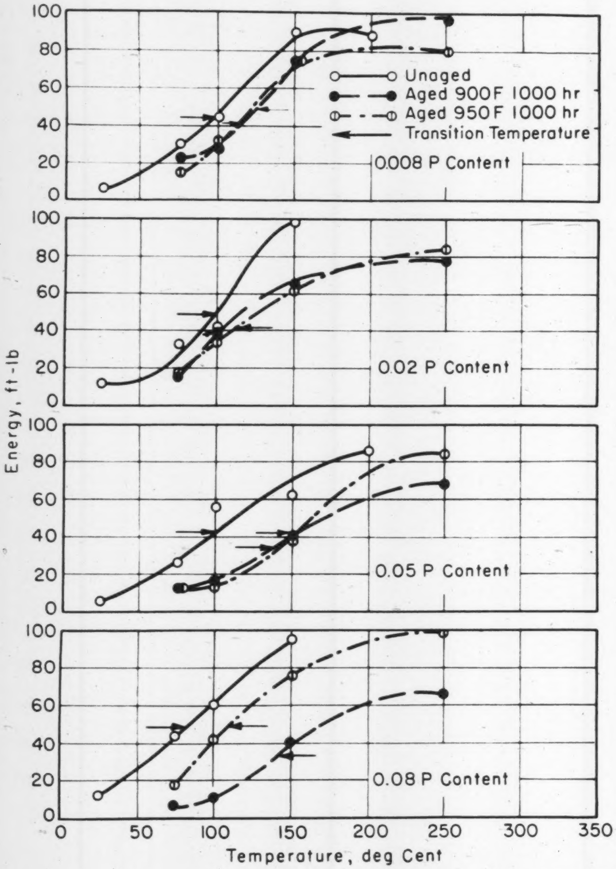


FIG. 4.—Effect of Phosphorus on the Energy Transition Temperature of a Cast Cr-Mo-V Steel.

tent of chromium, chromium-manganese and manganese steels resulted in a significant rise in the transition temperature of these steels both with and without an embrittling heat treatment.

*Furnace-Cooled Material:*

Twenty production heats of Cr-Mo-V steel were selected at random to establish the phosphorus content for the current basic electric melting practice of one of our suppliers. The results are presented in Table IV. The average phosphorus content was 0.011 per cent. Cr-Mo-V steel produced by the basic electric and open-hearth practice obtained from other suppliers ranges from 0.015 to 0.020 per cent phosphorus. The tempering cycle of cast Cr-Mo-V components includes a furnace cool from the tempering temperature. This cooling rate is approximately 20 C per hr.

TABLE III.—FRACTURE-APPEARANCE TRANSITION TEMPERATURES OF UNAGED AND AGED CAST Cr-Mo-V STEELS.

| Phosphorus Content, nominal weight per cent | Aging Temperature, deg Cent (deg Fahr) | Transition Temperature, deg Cent (deg Fahr) | Increase in Transition Temperature, deg Cent (deg Fahr) |
|---|---|---|---|
| 0.008.... | Unaged | 115 (239) | |
| 0.008.... | 482 (900) | 125 (257) | 10 (18) |
| 0.008.... | 510 (950) | 122 (252) | 7 (13) |
| 0.02..... | Unaged | 110 (230) | |
| 0.02..... | 482 (900) | 125 (257) | 15 (27) |
| 0.02..... | 510 (950) | 125 (257) | 15 (27) |
| 0.05..... | Unaged | 118 (244) | |
| 0.05..... | 482 (900) | 160 (320) | 42 (76) |
| 0.05..... | 510 (950) | 150 (302) | 32 (58) |
| 0.08..... | Unaged | 106 (223) | |
| 0.08..... | 482 (900) | 150 (302) | 44 (79) |
| 0.08..... | 510 (950) | 130 (266) | 24 (43) |

To simulate the above condition, Cr-Mo-V material at the 0.008 per cent phosphorus level was furnace cooled 20 C per hr from the tempering temperature. V-notch Charpy data are shown in Fig. 5 and summarized in Table V. The results show that, at the 0.008 per cent phosphorus level, material furnace cooled (20 C per hr) from the tempering temperature did not exhibit any significant rise in the transition temperature.

*Room Temperature Tensile Properties:*

A summary of the room temperature tensile properties is presented in Table VI and Fig. 6.

TABLE IV.—PHOSPHORUS ANALYSIS ON TWENTY HEATS OF BASIC ELECTRIC Cr-Mo-V STEEL.

| Heat | Phosphorus Content,[a] weight per cent | Heat | Phosphorus Content, weight per cent |
|---|---|---|---|
| No. 1404..... | 0.014 | No. 2042.... | 0.011 |
| No. 1407..... | 0.011 | No. 2046.... | 0.015 |
| No. 1413..... | 0.009 | No. 2048.... | 0.013 |
| No. 1414..... | 0.012 | No. 2050.... | 0.015 |
| No. 1436..... | 0.013 | No. 2062.... | 0.008 |
| No. 1456..... | 0.010 | No. 2068.... | 0.013 |
| No. 1468..... | 0.011 | No. 2091.... | 0.010 |
| No. 1471..... | 0.011 | No. 2097.... | 0.010 |
| No. 1490..... | 0.010 | No. 2099.... | 0.011 |
| No. 2022..... | 0.014 | No. 2108.... | 0.009 |

[a] Average phosphorus content 0.011 per cent.

(*a*) *Tensile Strength.*—In the range of 0.008 to 0.05 per cent phosphorus there is no general trend in the tensile strength of the unaged and aged material. At the 0.08 per cent phosphorus level the tensile strength decreases substantially for the unaged condition and steel aged at 950 F, whereas material aged at 900 F showed only a slight decrease.

(*b*) *Yield Strength.*—Of the levels investigated, the maximum yield strength resulted at the 0.008 per cent phosphorus content in the unaged and aged material. In general, increasing the phosphorus content from 0.008 to 0.08 per cent resulted in a decrease in the yield strength of both the unaged and aged material. This was due in part to

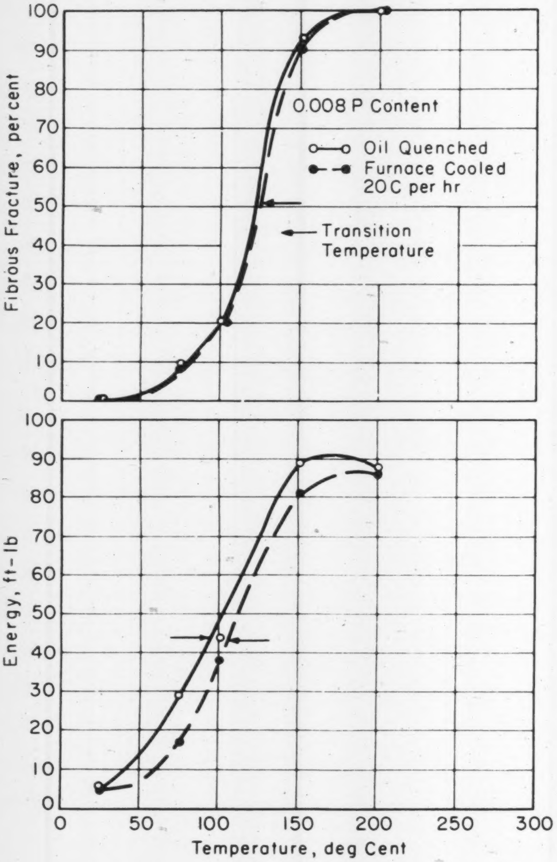


FIG. 5.—Energy and Fracture Appearance Transition Curves for Cast Cr-Mo-V Steel.

TABLE V.—V-NOTCH CHARPY RESULTS OF FURNACE COOLED AND OIL QUENCHED CAST Cr-Mo-V STEEL.

| Phosphorus Content, nominal weight per cent | Brinell Hardness Number[a] | ASTM Austenitic Grain Size | Heat Treatment | | 25 C (77 F) | 75 C (167 F) | 100 C (212 F) | 150 C (302 F) | 200 C (392 F) |
|---|---|---|---|---|---|---|---|---|---|
| 0.008..... | 228 | 3 to 4 | 1030 to 1075 C (1877 to 1967 F) for 12 hr, air cooled, 690 C (1275 F) for 17 hr, oil quenched | Energy, ft-lb...... | 6 | 29 | 44 | 89 | 88 |
| | | | | Per cent fibrous fracture........ | 0 | 10 | 20 | 93 | 100 |
| 0.008..... | 235 | 3 to 4 | 1030 to 1075 C (1877 to 1967 F) for 12 hr, air cooled, 690 (1275 F) for 14 hr, furnace cooled at 20 C per hr | Energy, ft-lb...... | 5 | 17 | 38 | 81 | 86 |
| | | | | Per cent fibrous fracture........ | 0 | 8 | 20 | 90 | 100 |

[a] Hardness of Charpy bars (average of 3 readings).

TABLE VI.—TENSILE PROPERTIES OF FOUR EXPERIMENTAL CAST Cr-Mo-V STEELS.

| Phosphorus Content, nominal weight per cent | Aging Temperature, deg Cent (deg Fahr) | Tensile Strength, psi | Yield Strength (0.02 per cent offset), psi | Elongation in 2 in., per cent | Reduction of Area, per cent | Quality Factor, TS × RA × $10^{-6}$ |
|---|---|---|---|---|---|---|
| 0.008 | Unaged | 108 500 | 85 000 | 19.0 | 61.8 | 6.7 |
| | | 108 500 | 83 750 | 18.0 | 51.4 | 5.5 |
| | 482 (900) | 89 000[a] | 77 500[a] | 3.0[a] | 5.8[a] | ... |
| | | 107 500 | 82 500 | 19.0 | 60.3 | 6.0 |
| | 510 (950) | 84 000[a] | 77 000[a] | 0[a] | 1.1[a] | ... |
| | | 111 500 | 83 750 | 18.5 | 54.1 | 6.0 |
| 0.02 | Unaged | 105 000 | 80 000 | 19.0 | 62.6 | 6.5 |
| | | 104 000 | 75 750 | 22.0 | 64.9 | 6.7 |
| | 482 (900) | 109 500 | 80 000 | 19.5 | 60.3 | 6.6 |
| | | 108 000 | 76 250 | 22.0 | 61.1 | 6.5 |
| | 510 (950) | 107 500[a] | 82 500[a] | 7.0[a] | 14.1[a] | ... |
| | | 87 000[a] | 68 750[a] | 1.5[a] | 2.3[a] | ... |
| 0.05 | Unaged | 110 000 | 80 000 | 20.0 | 57.5 | 6.3 |
| | | 107 500 | 77 500 | 23.0 | 56.2 | 6.0 |
| | 482 (900) | 110 000 | 77 500 | 18.5 | 48.6 | 5.3 |
| | | 110 000[a] | 80 000[a] | 10.0[a] | 21.3[a] | ... |
| | 510 (950) | 112 500 | 72 500 | 15.0 | 44.3 | 4.9 |
| | | 106 500 | 63 000 | 20.0 | 57.3 | 6.1 |
| 0.08 | Unaged | 101 500 | 73 000 | 22.0 | 56.8 | 5.7 |
| | | 97 500 | 71 750 | 16.0 | 35.7 | 3.4 |
| | 482 (900) | 109 500[a] | 75 000[a] | 12.5[a] | 29.5[a] | ... |
| | | 109 500 | 71 750 | 15.0 | 54.9 | 6.0 |
| | 510 (950) | 96 500 | 61 250 | 22.0 | 60.3 | 5.8 |
| | | 100 000 | 63 750 | 23.0 | 53.6 | 5.3 |

[a] Defective test material. Early shakeout resulted in localized hydrogen flaking.

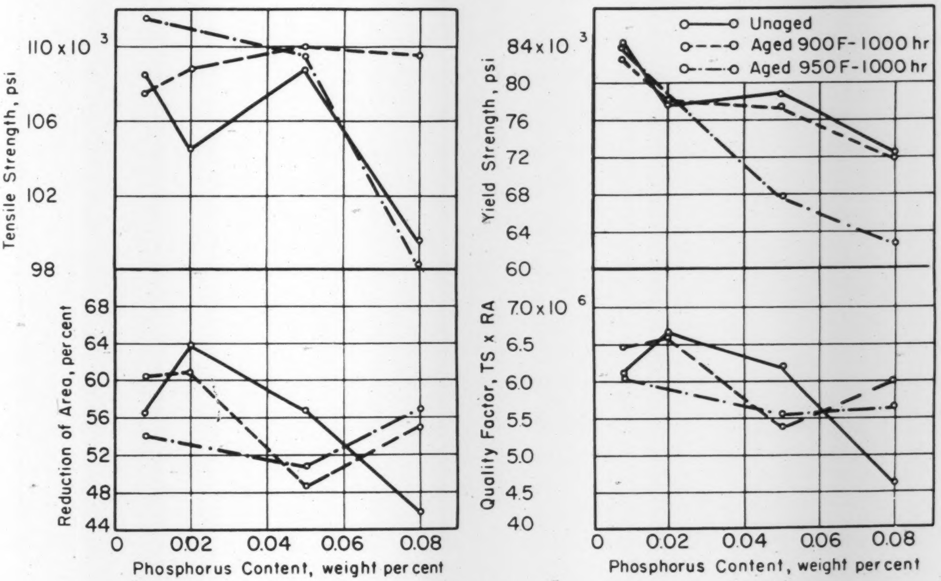


Fig. 6.—Effect of Phosphorus on Tensile Properties of a Cast Cr-Mo-V Steel.

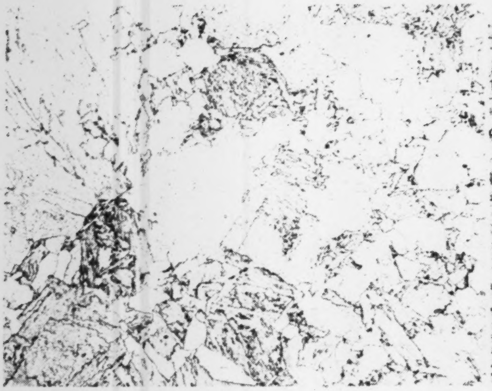


FIG. 7—0.008 per cent Phosphorus Content Unaged, Bainitic Matrix with Approximately 2 per cent Pro-Eutectoid Ferrite (× 100).

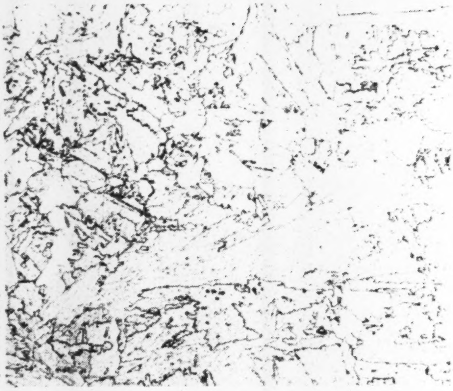


FIG. 8.—0.02 per cent Phosphorus Content Unaged, Bainitic Matrix with Approximately 5 per cent Pro-Eutectoid Ferrite (× 100).

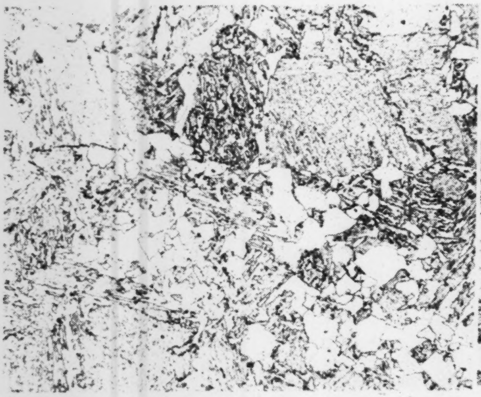


FIG. 9.—0.05 per cent Phosphorus Content Unaged, Bainitic Matrix with Approximately 5 per cent Pro-Eutectoid Ferrite (× 100).

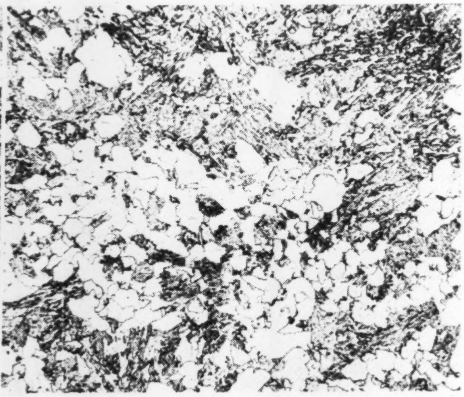


FIG. 10.—0.08 per cent Phosphorus Content Unaged, Bainitic Matrix with Approximately 40 per cent Pro-Eutectoid Ferrite (× 100).

the increase of pro-eutectoid ferrite at the higher phosphorus levels.

(*c*) *Tensile Ductility and Quality Factor* (*TS* × *RA*).—For the material unaged and aged at 900 F the maximum tensile ductility and quality factor occur at the 0.02 per cent phosphorus level. The quality factor is defined as the product of tensile strength and per cent reduction of area. Beyond 0.02 per cent, the unaged material exhibits a decrease in tensile ductility and quality factor with increasing phosphorus. In general, increasing phosphorus from 0.02 to 0.05 per cent showed a decrease in the tensile ductility and quality factor of materials aged at 900 and 950 F. A further increase of phosphorus from 0.05 to 0.08 per cent resulted in an increase in tensile ductility and quality factor. The increase in these properties may be attributed to the higher pro-eutectoid ferrite content at the 0.08 per cent phosphorus level.

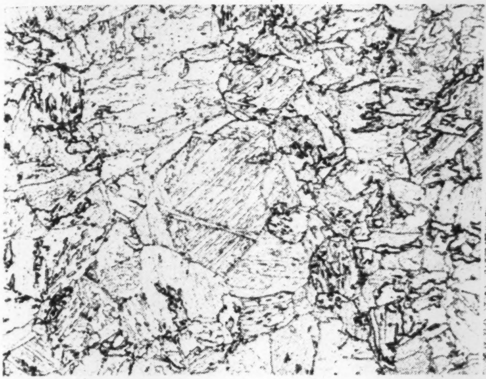


Fig. 11.—0.008 per cent Phosphorus Content, Aged at 900 F for 1000 hr, Bainitic Matrix Approximately 2 per cent Pro-Eutectoid Ferrite (× 100).

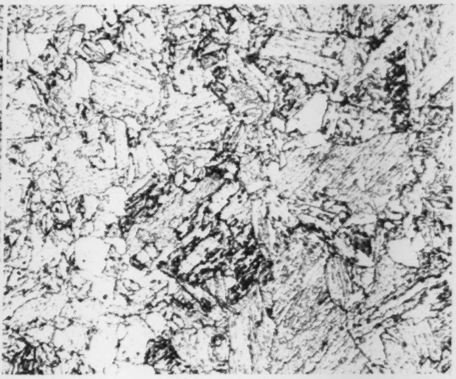


Fig. 12.—0.02 per cent Phosphorus Content, Aged at 900 F for 1000 hr, Bainitic Matrix Approximately 5 per cent Pro-Eutectoid Ferrite (× 100).

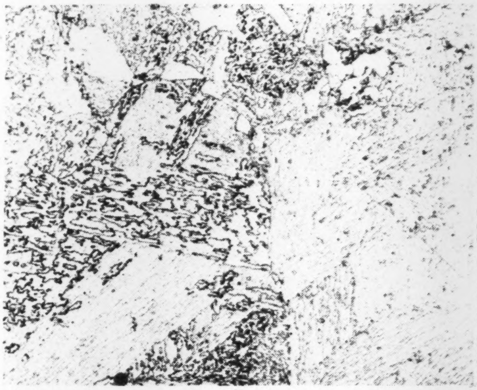


Fig. 13.—0.05 per cent Phosphorus Content, Aged at 900 F for 1000 hr, Bainitic Matrix less than 5 per cent Pro-Eutectoid Ferrite (× 100).

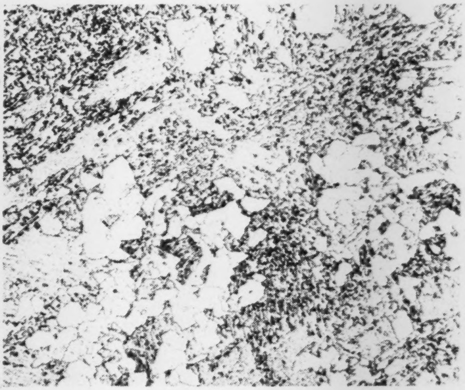


Fig. 14.—0.08 per cent Phosphorus Content, Aged at 900 F for 1000 hr, Bainitic Matrix Approximately 20 per cent Pro-Eutectoid Ferrite (× 100).

*Microstructure:*

Representative microstructures of the unaged and aged material are shown and described in Figs. 7 to 18. In the unaged and aged materials only a slight increase in pro-eutectoid ferrite was noted up to the 0.05 per cent phosphorus level. However, a substantial increase was noted at the 0.08 per cent phosphorus level. The increase in pro-eutectoid ferrite may be due to the increased phosphorus content. Also, it is possible that the 0.08 per cent phosphorus content ingot received a slower cool during the normalize.

To determine the cause of the increased ferrite, additional material at the 0.05 and 0.08 per cent phosphorus level was re-austenitized in the laboratory at 1050 C (1920 F) for 12 hr and air-cooled. Specimens of each material were removed from an identical position for microscopic investigation. Representative microstructures are shown and

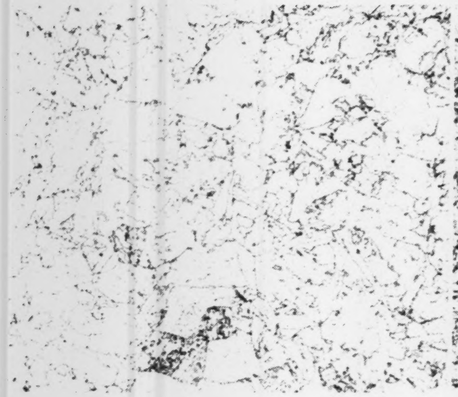


FIG. 15.—0.008 per cent Phosphorus Content, Aged at 950 F for 1000 hr, Bainitic Matrix Approximately 2 per cent Pro-Eutectoid Ferrite (× 100).

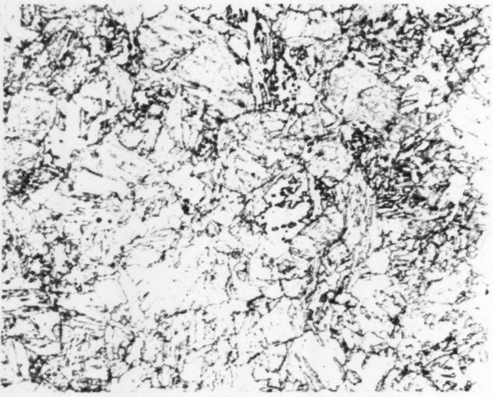


FIG. 16.—0.02 per cent Phosphorus Content, Aged at 950 F for 1000 hr, Bainitic Matrix Approximately 2 per cent Pro-Eutectoid Ferrite (× 100).

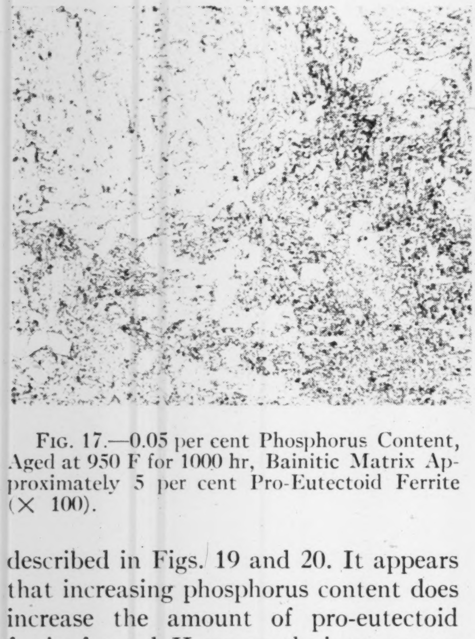


FIG. 17.—0.05 per cent Phosphorus Content, Aged at 950 F for 1000 hr, Bainitic Matrix Approximately 5 per cent Pro-Eutectoid Ferrite (× 100).

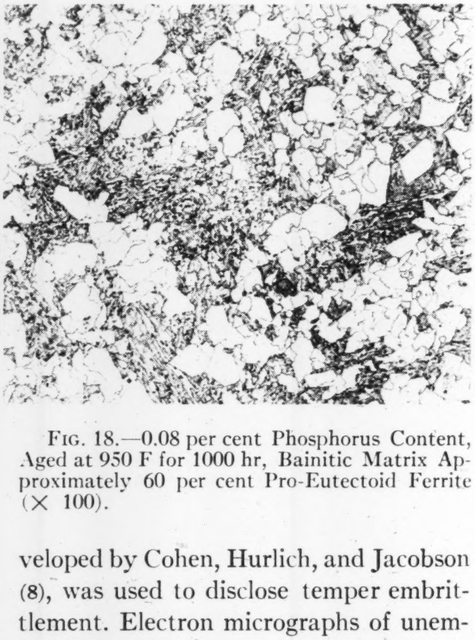


FIG. 18.—0.08 per cent Phosphorus Content, Aged at 950 F for 1000 hr, Bainitic Matrix Approximately 60 per cent Pro-Eutectoid Ferrite (× 100).

described in Figs. 19 and 20. It appears that increasing phosphorus content does increase the amount of pro-eutectoid ferrite formed. However, the increase was only 10 per cent instead of the previously estimated 40 per cent. Phosphorus *per se* appears to lower the ferritic hardenability of Cr-Mo-V steel and the effect is most pronounced above 0.05 per cent phosphorus.

A Zepherian-chloride reagent, developed by Cohen, Hurlich, and Jacobson (8), was used to disclose temper embrittlement. Electron micrographs of unembrittled and embrittled material are shown in Figs. 21 and 22. The embrittled specimen exhibited a groove formation at the former austenitic grain boundaries indicating temper embrittlement, whereas no evidence of groove formation is shown in the unaged material.

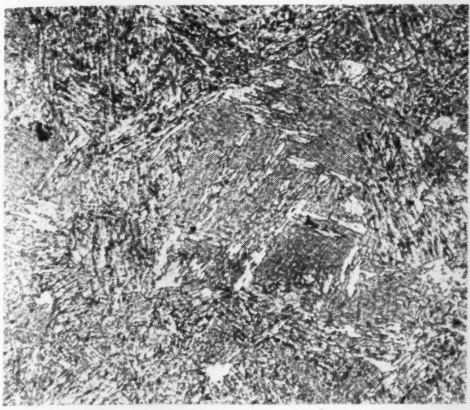


FIG. 19.—0.05 per cent Phosphorus Content, Unaged, Re-Austenitized, Bainitic Matrix with Less than 2 per cent Pro-Eutectoid Ferrite (× 100).

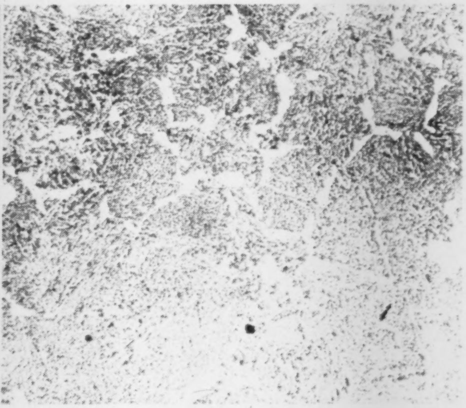


FIG. 20.—0.08 per cent Phosphorus Content, Unaged, Re-Austenitized, Bainitic Matrix with Approximately 10 per cent Pro-Eutectoid Ferrite (× 100).

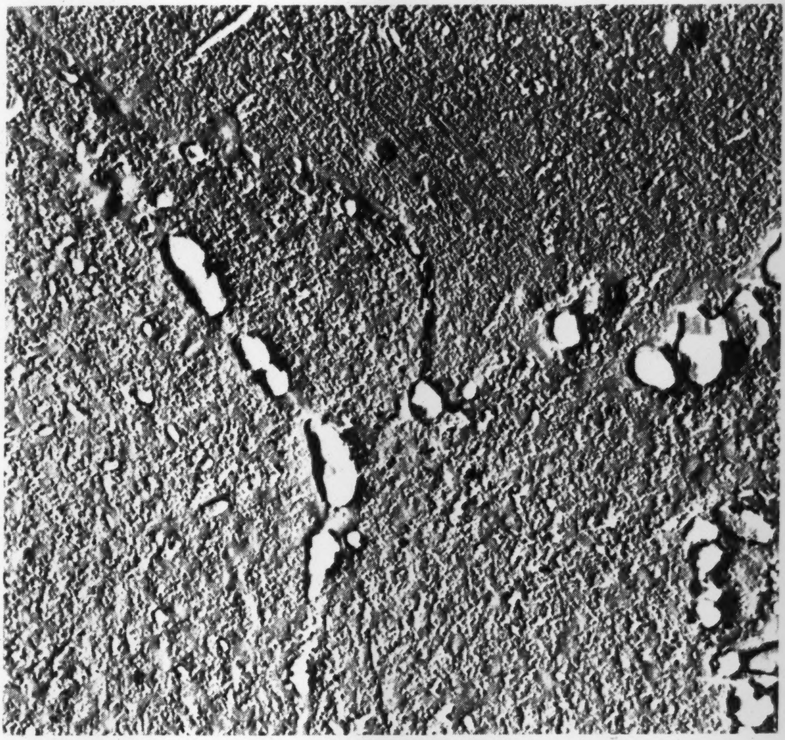


FIG. 21.—0.08 per cent Phosphorus Content, Unaged, No Evidence of Temper Embrittlement, Zepherian-Chloride Etchant (× 10,000).

## CONCLUSIONS

The effect of phosphorus content on the impact and tensile properties and susceptibility to temper embrittlement of a nominal 0.20 per cent carbon, 1 chromium, 1 molybdenum, 0.2 vanadium cast steel was determined. The following conclusions are drawn:

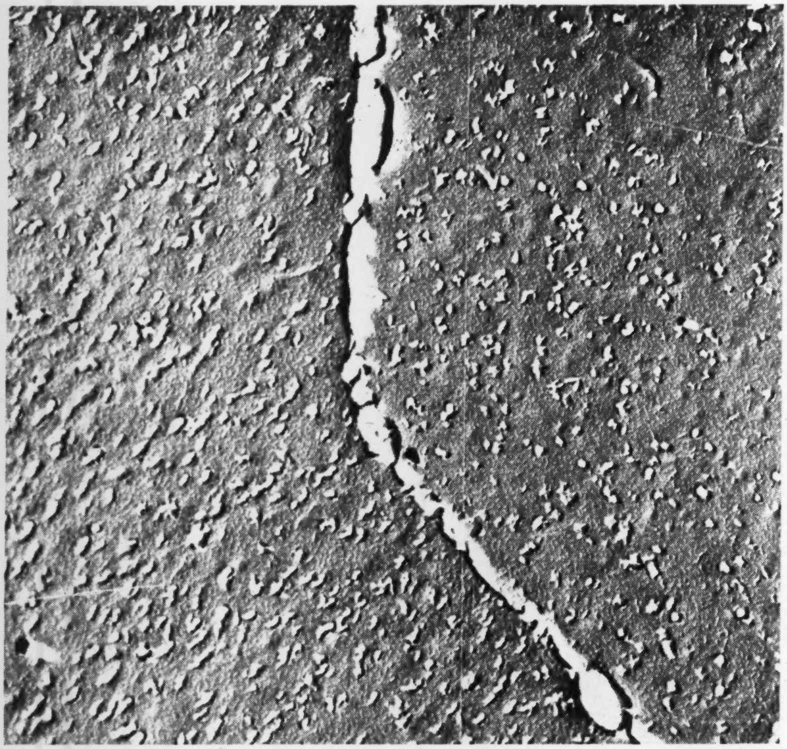


FIG. 22.—0.08 per cent Phosphorus Content, Aged at 900 F for 1000 hr, Evidence of Groove Formation Indicating Temper Embrittlement, Zepherian-Chloride Etchant (× 10,000).

1. Cast Cr-Mo-V steels with phosphorus contents of 0.05 and 0.08 per cent were susceptible to temper embrittlement as indicated by the marked rise in the fracture-appearance transition temperature.

2. Cast Cr-Mo-V steels with 0.008 and 0.02 per cent phosphorus content, isothermally aged at 900 and 950 F, showed only slight evidence of temper embrittlement.

3. Of the phosphorus contents investigated, the optimum room temperature tensile properties were exhibited at the 0.008 and 0.02 per cent phosphorus level.

4. The notch-bar toughness of the unaged cast Cr-Mo-V material, oil quenched from the temper, is not significantly affected by an increase of phosphorus from 0.008 to 0.08 per cent.

5. Increasing phosphorus from 0.008 to 0.08 per cent results in an increase in the amount of pro-eutectoid ferrite by approximately 10 per cent. Phosphorus *per se* appears to lower the ferritic hardenability of cast Cr-Mo-V steel.

*Acknowledgment:*

The authors wish to express their appreciation to A. E. Powers, Materials and Processes Laboratory, Large Steam Turbine-Generator Dept., General Electric Co., for his helpful discussions and advice.

## REFERENCES

(1) A. Preece and R. D. Carter, "Temper Brittleness in High-Purity Iron-Base Alloys," *Journal*, Iron and Steel Inst., London, Vol. 173, Apr., 1953, pp. 387-398.

(2) H. Jolivet and G. Vidal, "Importance of the Impact Test for Study of Temper Brittleness," *Revue de Metallurgie*, Vol. 41, pp. 378-388, 403-408 (1944).

(3) R. Hultgren and J. Chuan Chang, "Investigation on the Temper Brittleness of Steels," *Final Report No. N6-ori-211*, University of California, Feb. 15, 1951.

(4) A. E. Powers, "The Influence of Molybdenum and Tungsten on Temper Brittleness," *Transactions*, Am. Soc. Metals, Vol. 48, pp. 149-164 (1956).

(5) M. Baeyertz, W. F. Craig, Jr., and J. P. Sheehan, "The Effects of Molybdenum and Commercial Ranges of Phosphorus upon the Toughness of 0.40 per cent Carbon Chromium Steels," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 185, pp. 535-543 (1949).

(6) B. C. Woodfine, "Some Aspects of Temper-Brittleness," *Journal*, Iron and Steel Inst., London, Vol. 173, March 1953, pp. 240-255.

(7) S. A. Herres and A. R. Elsea, "Investigation of Temper Brittleness in Low-Alloy Steels," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 185, pp. 366-370 (1949).

(8) J. B. Cohen, A. Hurlich, and M. Jacobson, "A Metallographic Etchant to Reveal Temper Brittleness," *Transactions*, Am. Soc. Metals, Vol. 36, pp. 473-540 (1946).

## DISCUSSION

MR. JOHN M. HODGE.[1]—Does the author ascribe this so-called temper brittleness to the classical temper brittleness or is this a case of alloy carbide precipitation and embrittlement from this precipitation.

I would also like to know whether this behavior shows up in the elevated temperature ductility or in the stress rupture strength?

MR. ROBERT S. ZENO (*author*).—First we wanted to avoid getting into any theories on temper embrittlement because there are many and one could take his choice of any one of them. We feel that we found classical temper embrittlement of a reversible variety (as measured by loss in toughness). The Cr-Mo-V material tested has lost toughness as measured by a rise in the energy or fracture appearance transition temperature.

On the second part of your question, by rupture ductility—I assume you are talking about either an elongation or reduction of area at fracture. The rupture strength and rupture ductility were not affected at the lower phosphorus levels (up to 0.030 per cent). We have no data on higher phosphorus contents.

MR. HODGE.—What is the evidence for this reversibility?

MR. ZENO.—The evidence we have is the result of isothermal aging treatments on this particular composition and similar cast chromium-molybdenum and chromium-molybdenum-vanadium compositions where the chrome and the moly are slightly different. We aged these materials at 850, 900, 950, 1000, 1050, and 1100 F. The only transition temperature rise that we have observed due to temper embrittlement was in the 900 and 950 F range. If the same embrittled material is heated up to temperatures above this, the original toughness (transition temperature) is restored. Based on these results we feel that the temper embrittlement in these alloys is reversible.

MR. HARRY W. HIGHRITER.[2]—It may

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[1] Chief Research Engineer, Alloy-Steel, U. S. Steel Corp., Pittsburgh, Pa.

[2] Vice-president and Technical Director, Fansteel Metallurgical Corp., North Chicago, Ill.

be of interest to point out what seems to me a most interesting parallel of the phenomena reported with some work which was done a number of years ago on malleable cast iron relative to the embrittlement by the hot-dip galvanizing process.

I think it was in 1930 that W. R. Bean reported and published in the *Transactions* of the American Foundrymen's Assn. a paper which summarized the work which E. S. Davenport and I did shortly prior to that time. We found that the embrittlement of malleable iron castings, when these were galvanized by the hot-dip process, was definitely related to the phosphorus and silicon contents. The permissible maximum limits for each of these elements were interrelated so that if one were high the other had to be below a certain value. In the manufacture of malleable castings, the malleablizing heat treatment which converts the product from the initial white iron castings requires several days and would correspond to the aging reported in the current paper.

It was found by Marshall, then of the Bureau of Standards and who published and patented the process, that the galvanizing embrittlement could be prevented by a preliminary heat treatment which consisted of quenching in water at room temperature castings heated to about 1200 F. Under these conditions, the limits for phosphorus and silicon which we had imposed were substantially altered. This preventive heat treatment would confirm the reversibility reported in the current paper.

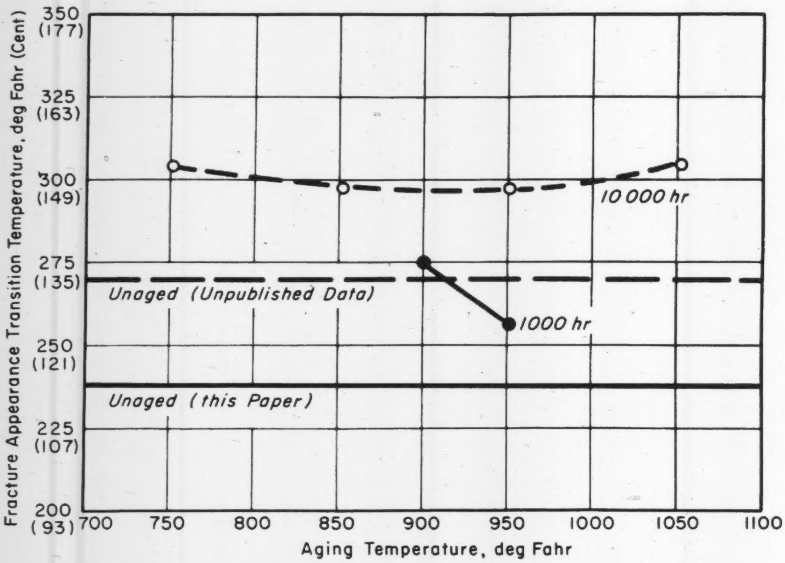

FIG 23.—Transition Temperature as a Function of Aging Temperature and Time for Cast 1 Chromium, 1 Molybdenum, $\frac{1}{4}$ Vanadium Steel Containing 0.027 Phosphorus.

MR. ZENO.—Additional data obtained on a cast 1 per cent chromium, 1 molybdenum, $\frac{1}{4}$ vanadium steel containing 0.027 per cent phosphorus further substantiates the results of this paper. Note the rise in transition temperature for the material aged at various temperatures for 10,000 hr (Fig 23). The need for phosphorus control in this family of alloys has been further demonstrated.

# THE STATIC PROPERTIES OF SEVERAL HIGH-STRENGTH STEELS*

By E. P. Klier,[1] B. B. Muvdi,[1] and G. Sachs[1]

The need for light-weight component construction, particularly in the aircraft industry has led to the use of low-alloy steels heat treated to strengths in the range above 200,000 psi. Steels in this strength range, however, tend to be notch sensitive, and this factor must be suitably weighed in the selection and application of these high-strength materials.

Notch properties of high-strength steels are known to depend on (*a*) strength level, (*b*) orientation, (*c*) composition, (*d*) eccentricity of the load, (*e*) as-processed section size; and (*f*) stress concentration. Also of importance are the test specimen size and the variability of properties due to composition changes within a given type of steel (**1, 2, 3**).[2]

In the present paper are presented the preliminary results of an investigation of the influence of the above factors on the notch tension properties of ten steels heat treated to strengths above 200,000 psi. In obtaining the data, tension, notch-tension (concentric and eccentric loading) and stress-rupture tests were completed.

## Experimental Procedure

### *Materials:*

The steels investigated were four heats of 4340, vanadium-modified 4330, 98B40, and the proprietary steels Hy-Tuf, Crucible UHS-280, Super TM-2, and Tricent. The chemical compositions and bar sizes of these materials are given in Table I.

### *Test Specimens:*

The test specimens are shown in Fig. 1. The specimens were rough machined to within +0.015 in. of the final dimension on each side, heat treated as indicated in the respective figures and then finish machined. Specimens oriented parallel (longitudinal) and perpendicular (transverse) to the rolling direction were investigated. Both 0.3- and 0.5-in. diameter notch specimens were tested as indicated. The respective specimen dimensions were suitably scaled to give the appropriate theoretical stress concentrations.

### *Tension Tests:*[3]

The specimen shown in Fig. 1 (*a*) was used for vanadium modified 4330. Head breaks for transverse specimens were experienced for certain of the other steels and the stepped specimen (Fig. 1, (*b*)) was, therefore, used for all steels except the vanadium modified 4330.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Professor of Metallurgical Engineering, Research Associate and Associate Director of Research, respectively, Syracuse University Research Institute, Syracuse, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 727.

[3] Axiality of load application was ensured by the use of suitable fixtures which have essentially been described elsewhere.

*Notch Tension Tests:*[3]

Theoretical stress concentration factors of 3, 5, and 10 were obtained for both the 0.3- and 0.5-in. diameter specimens. The notch dimensions to produce these stress concentrations were determined by Neuber's theory according to Peterson (4).

TABLE I.—INFORMATION PERTINENT TO THE STEELS INVESTIGATED.

| Alloy | Shape and Size | Alloying Elements, per cent | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Carbon | Manganese | Phosphorous | Sulfur | Silicon | Nickel | Chromium | Molybdenum | Vanadium | Copper |
| 4340, heat No. 1 | 4¼ in. round<br>1½ in. round | 0.41 | 0.79 | 0.013 | 0.016 | 0.31 | 1.83 | 0.77 | 0.23 | ... | ... |
| 4340, heat No. 2 | 4 in. round | 0.40 | 0.74 | 0.023 | 0.029 | 0.19 | 1.83 | 0.77 | 0.24 | ... | ... |
| 4340, heat No. 3 | 3½ in. round | 0.425 | 0.83 | 0.014 | 0.013 | 0.30 | 1.77 | 0.80 | 0.24 | ... | ... |
| 4340, heat No. 4 | 3¼ in. round | 0.415 | 0.75 | 0.011 | 0.014 | 0.31 | 1.76 | 0.81 | 0.24 | ... | ... |
| Vanadium modified 4330 | 4 in. square | 0.32 | 0.88 | 0.012 | 0.018 | 0.26 | 1.79 | 0.84 | 0.355 | 0.07 | ... |
| 98B40 | 4½ in. round | 0.46 | 0.79 | 0.017 | 0.017 | 0.35 | 0.86 | 0.81 | 0.19 | ... | ... |
| Hy-Tuf | 3 in. round | 0.285 | 1.29 | 0.019 | 0.015 | 1.58 | 1.87 | 0.24 | 0.40 | ... | ... |
| Crucible UHS-280 | 3 in. round | 0.41 | 1.28 | 0.014 | 0.024 | 1.77 | | 1.26 | 0.33 | 0.17 | ... |
| Super TM-2 | 3 in. round | 0.41 | 0.72 | 0.012 | 0.014 | 0.61 | 2.08 | 1.15 | 0.44 | ... | 0.14 |
| Tricent | 4½ in. square | 0.39 | 0.74 | 0.014 | 0.014 | 1.54 | 1.83 | 0.83 | 0.38 | 0.07 | ... |

*Eccentric Notch Tension Tests:*

Longitudinal specimens, 0.3 in. in diameter, were tested at stress concentrations of $K = 3$, 5, and 10. The load was applied on a line through the root of the notch by means of a suitable fixture (5).

*Sustained Load Tests:*

Longitudinal 4340 steel specimens, 0.3 in. in diameter, were tested under sustained loads at room temperature. Stress concentrations of $K = 1$ and 5 were employed.

## EXPERIMENTAL RESULTS—4340 STEEL

*Hardenability Tests:*

Aircraft quality 4340 steel, among other requirements, must fall within the limits of the AISI hardenability band. Since the specified hardenability band is relatively wide, property changes may be associated with hardenability changes within the hardenability spread for the steel. To determine if such a relationship exists, the testing for 4340 steel was completed on four heats of the steel. The hardenability data for these steels are presented in Fig. 2 and are seen to characterize three high-(heats 1, 3, and 4) and one low-(heat 2) hardenability heats. Furthermore, Fig. 2 includes hardenability data for an additional heat (heat 5) which was previously studied at Syracuse University (6).

*Tension Properties Variations:*

The tension test data for the 4340 steels are presented in Fig. 3. Specimens for the longitudinal direction, Fig. 3 (*a*), were obtained from the five heats with the hardenabilities indicated in Fig. 2. In addition, the as-processed section size of these materials varied from $\frac{7}{8}$ to $4\frac{1}{4}$ in. in diameter. Despite the differences in hardenability and as-processed section size, no marked variations in the tensile properties were observed for longitudinal specimens.

Comparative transverse tests were

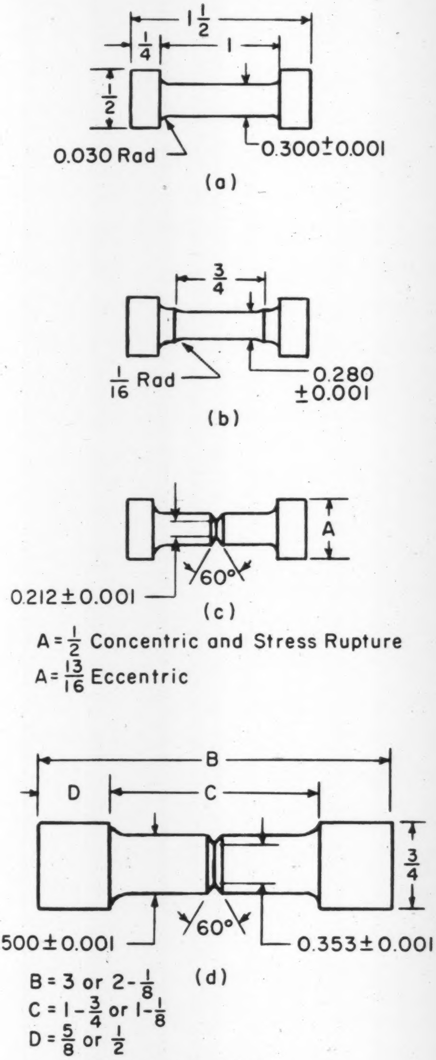

FIG. 1.—Test Specimens.

(*a*) 0.3-in. diameter tension specimen.
(*b*) 0.28-in. diameter tension and stress rupture specimen
(*c*) 0.3-in. diameter notch-tension specimen.
(*d*) 0.5-in. diameter notch-tension specimen.

completed except for the $\frac{7}{8}$-in. diameter bar stock. The results are presented in Fig. 3 (*b*). The strength properties compared favorably with those measured in the longitudinal direction. The ductility, however, as indicated by elongation and reduction of area, was reduced in the transverse direction. In general, the transverse reduction of area decreased as the as-processed section size was reduced.

*Notch-Strength Variations Produced by Changes in Test Conditions:*

The notch strength of a high-strength steel is modified by the stress concentration, specimen orientation, and specimen size. The influence of these factors on the notch strength of 4340 steel as the tensile strength was varied is given in Fig. 4. In general, as the tensile strength increased above 200,000 psi the sensitivity to notching was enhanced. As the stress concentration was increased, notch sensitivity increased. As the specimen orientation was changed from parallel to transverse to the rolling direction, the notch strength was profoundly lowered. Finally, as the diameter of the test section increased while the specimen geometry was adjusted to retain the theoretical stress concentration constant, notch sensitivity was enhanced.

*Notch-Strength Variations:*

The notch strength of 4340 steel from five heats is plotted in Fig. 5, as a function of strength level, stress concentration, and specimen orientation.

The notch strength for longitudinal specimens, Fig. 5 (*a*), scattered about a fairly well defined mean value for the different heats of 4340 steel. Only minor variations in the data were observed.

The notch strength for transverse specimens, Fig. 5 (*b*), also scattered about a fairly well defined mean value as long as the as-processed section sizes of the compared heats were about the same. However, as the as-processed section size was reduced, the transverse notch strength was also reduced. This effect was observed to increase with increase in the stress concentration.

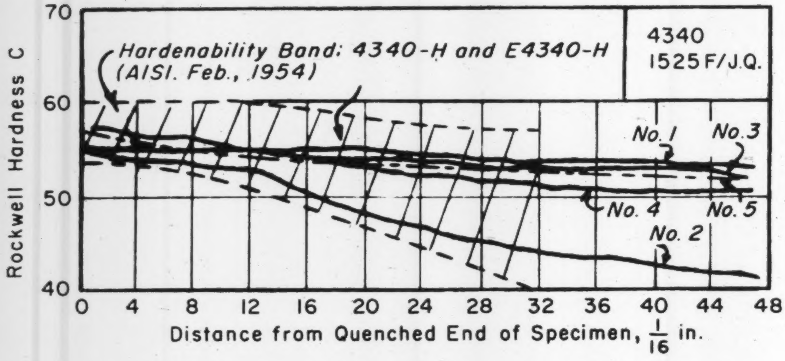

FIG. 2.—Jominy Hardenability of Various Heats of 4340 Steel.

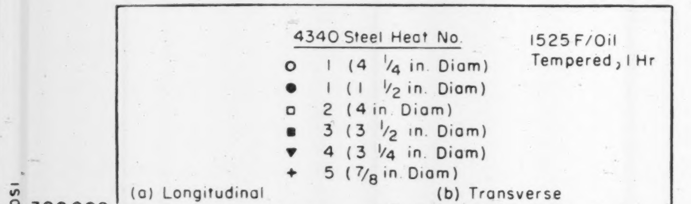

FIG. 3.—Effect of Tempering Temperature on the Strength and Ductility of 4340 Steel.

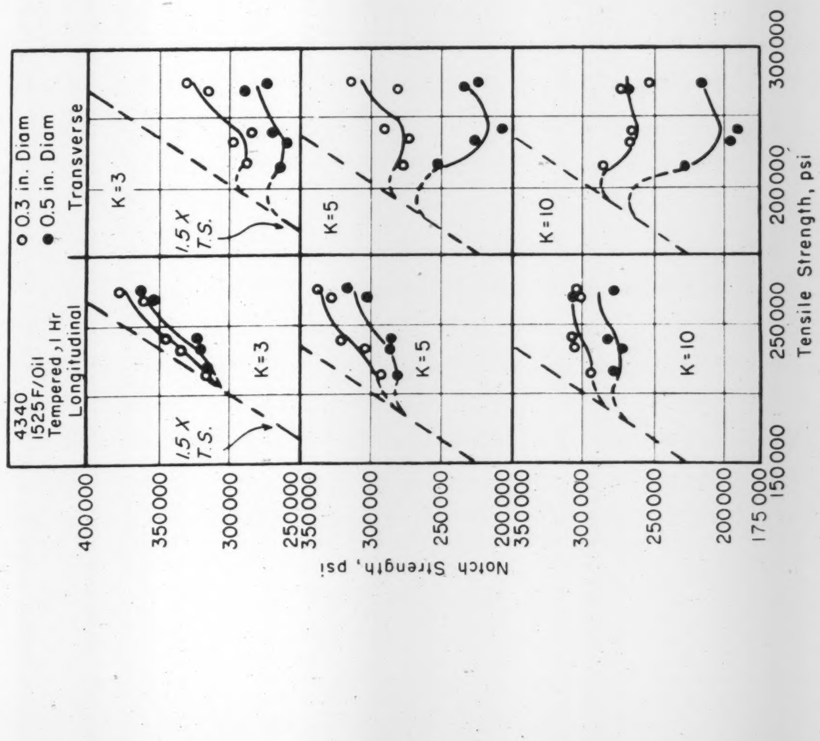

FIG. 4.—Notch Strength for 0.3 and 0.5 In. Diameter Longitudinal and Transverse Specimens from 4340 Steel (Heat No. 1—4¼ in. Diameter).

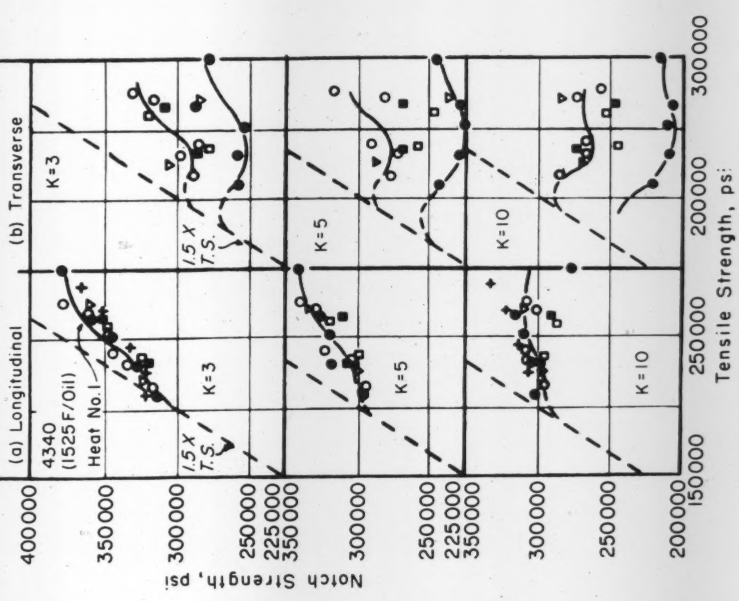

FIG. 5.—Notch Strength of Four Heats of 4340 Steel as a Function of Tensile Strength for 0.3-in. Diameter Specimens.

*Notch-Strength and Eccentric Loads:*

Although concentric-notch tests yield valuable information relating to the behavior of constructional steels in the presence of stress raisers, actual service conditions to which a steel part may be subjected are not well simulated by this test. A steel part with an irregular contour is usually subjected, in service, to a number of combinations of loads which may result in a complex state of stress and strain. To simulate such service conditions the eccentric-notch test has been proposed (7,8). This test differs from the concentric-notch test in that the line of load application is offset by a certain distance from the specimen centerline. In the eccentric tests performed in the present investigation, the eccentricity was approximately 0.106 in., this value being selected so that the line of load application passed close to the root of the notch.

The results of the eccentric notch-tension tests that were performed on four heats of 4340 steel are presented in Fig. 6. These results clearly reveal the pronounced reduction in notch-strength due to eccentricity, to values as low as 35 to 45 per cent of the concentric notch strength. The change in the notch strength due to the introduction of eccentric loading was far more important than that arising from the change in stress concentration.

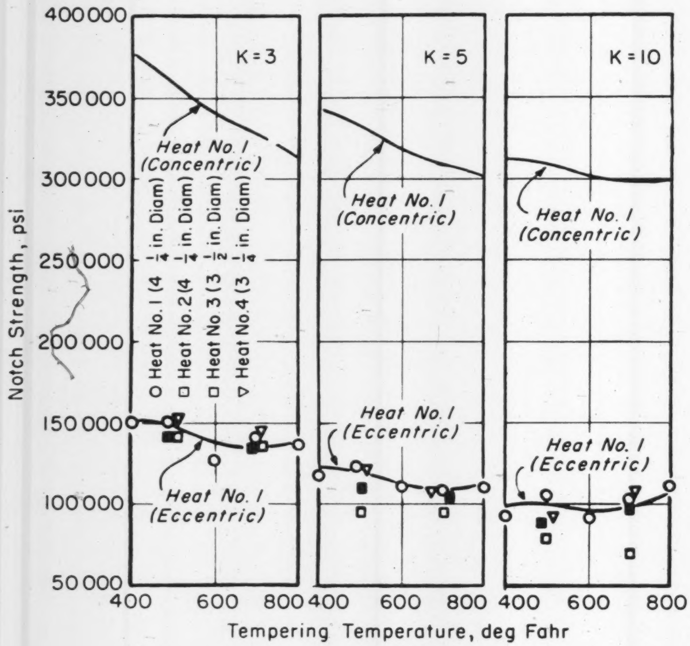

FIG. 6.—Concentric and Eccentric ($e$ = 0.106 in.) Notch Strength of Four Heats of 4340 Steel as a Function of Tempering Temperature for 0.3-in. Diameter Longitudinal Specimens.

*Strength of Specimens Under Sustained Load:*

It has recently been proposed that sustained loads may promote failure in high-strength steels due to "static fatigue" (9,10). Static fatigue is here used to signify fracturing due to the action of hydrogen which may be either residual or introduced in cleaning and plating operations. Limited testing was completed to determine if sustained load failures were to be expected for the commercial 4340 steel under investigation, and the results are presented in Fig. 7.

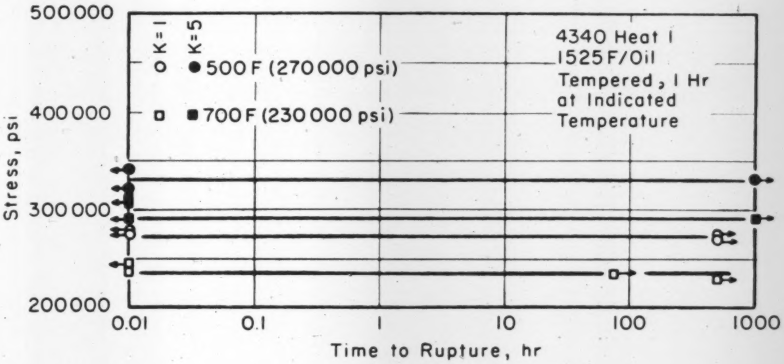

FIG. 7.—Stress-Rupture Diagram for Smooth and Notched 4340 Specimens at Two Strength Levels.

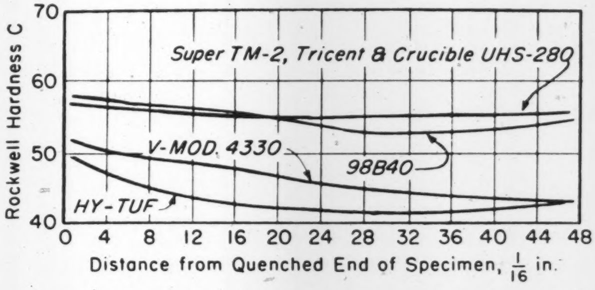

FIG. 8.—Jominy Hardenability of a Number of High-Strength Steels.

Both smooth and notched ($K = 5$) specimens were loaded to a value within the determined scatter range of the strengths for the selected specimens. These specimens either broke immediately on loading or remained unbroken at the end of the time given in Fig. 7.

From these data, it is concluded that sustained load failures in high-strength steels result from the action of hydrogen and are not due to static fatigue as is experienced in glasses and glasslike materials.

## ADDITIONAL HIGH-STRENGTH STEELS

### *Hardenabilities:*

The several steels which have been proposed for applications in the strength range above 200,000 psi invariably possess deep hardenability as indicated in the standard Jominy test, Fig. 8. However, the Jominy hardenability as measured for such deep-hardening steels may not give an adequate indication of their relative response when quenched in large sections (**11, 12**).

### *Tensile and Yield Strengths:*

The tensile and yield strengths *versus* tempering temperature for the several additional steels studied are presented in Fig. 9 for the longitudinal direction and in Fig. 10 for the transverse direction. Based on the tensile strength variations with tempering temperature, two groups of steels are given, namely, (*a*) that in which a range in strengths is possibly depending on the tempering temperature, for example vanadium modified 4330, 98B40, and super TM-2, and (*b*) that in which, due probably to a secondary hardening effect, a fairly constant tensile strength is maintained over much of the practical tempering temperature range, for example, Hy-Tuf, Crucible UHS-280 and Tricent. For the steels studied, in the aggregate, tensile strengths ranging from about 200,000 to 300,000 psi may readily be obtained.

The yield strengths for all materials tend to parallel the tensile strength as the tempering temperature is varied, except at the lowest tempering temperatures. In general, as the tempering temperature was reduced to the lowest value studied, the yield strength trend was to lower values.

For none of the steels studied was there a significant relationship between the orientation of the specimen and the tensile and yield strengths. On the other hand, for all materials, the transverse ductility, as measured by the reduction of area, was relatively reduced.

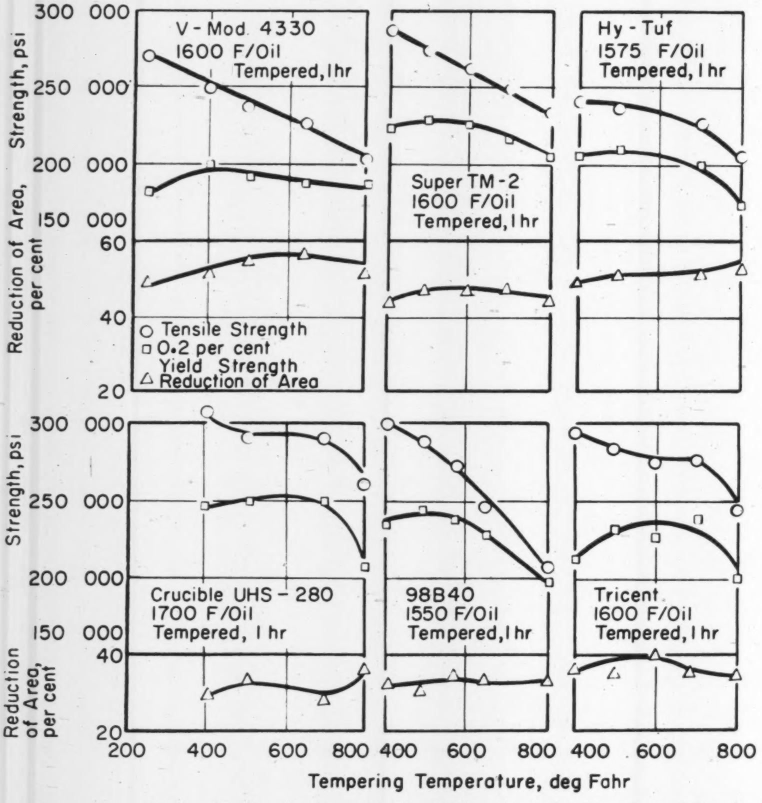

FIG. 9.—Strength and Ductility in the Longitudinal Direction of a Number of Steels as a Function of Tempering Temperature.

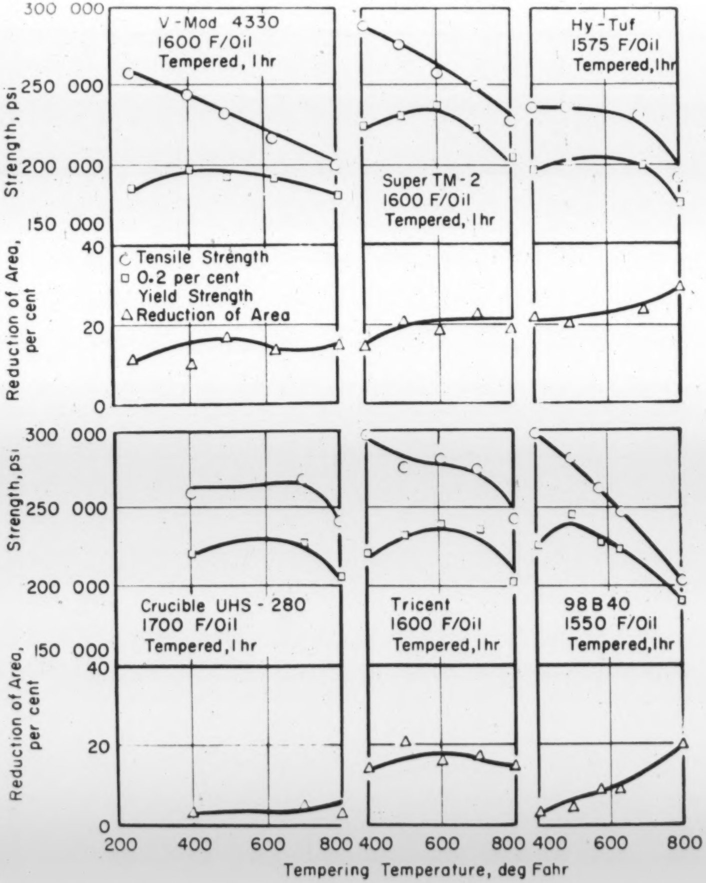

FIG. 10.—Strength and Ductility in the Transverse Direction of a Number of Steels as a Function of Tempering Temperature.

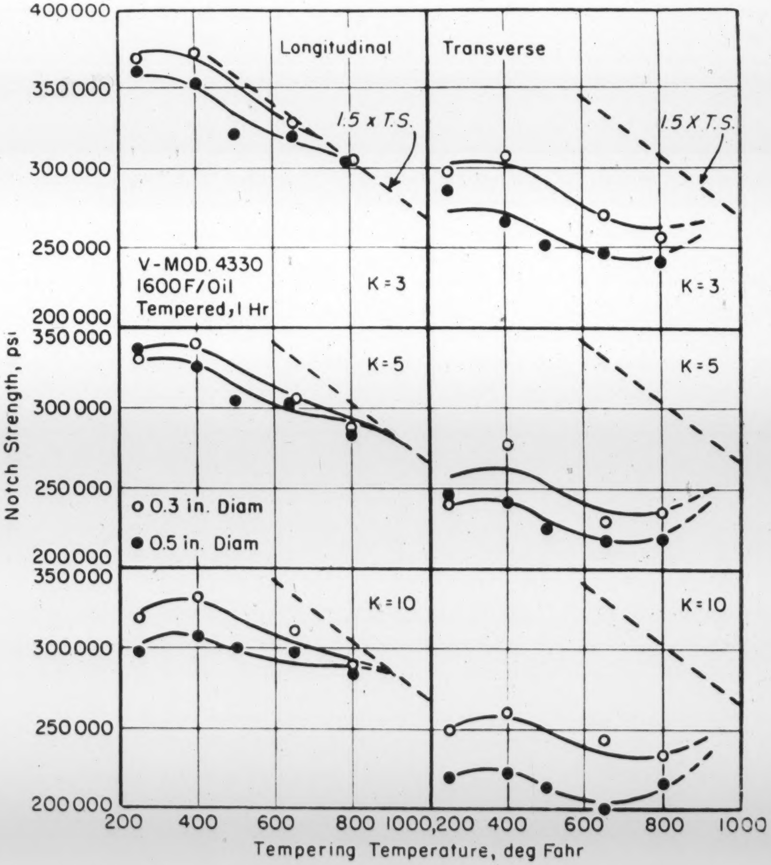

FIG. 11.—Notch Strength of Vanadium Modified 4330 Steel as a Function of Tempering Temperature.

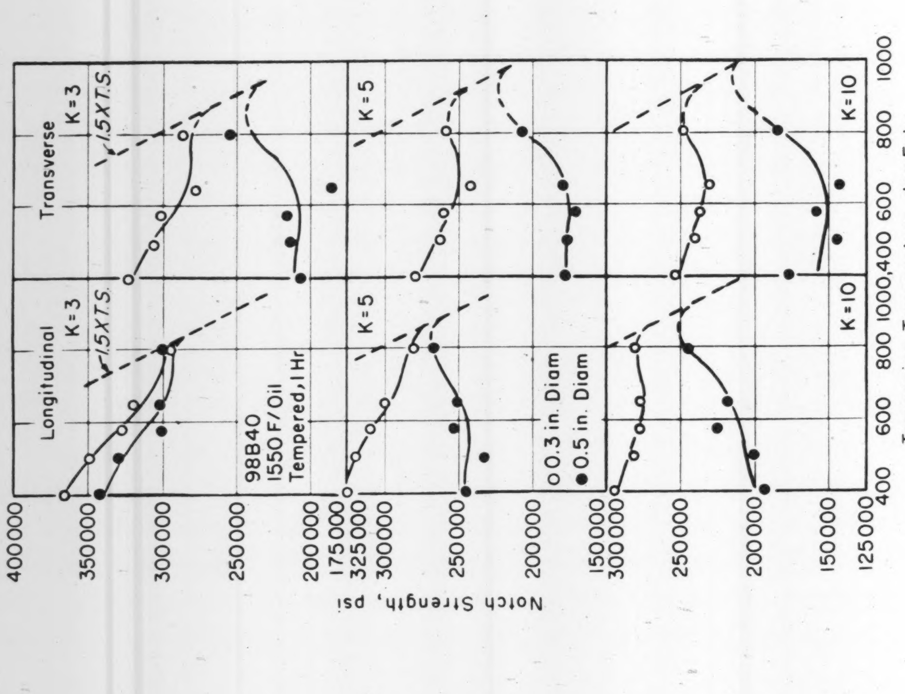

FIG. 12.—Notch Strength of 98B40 Steel as a Function of Tempering Temperature.

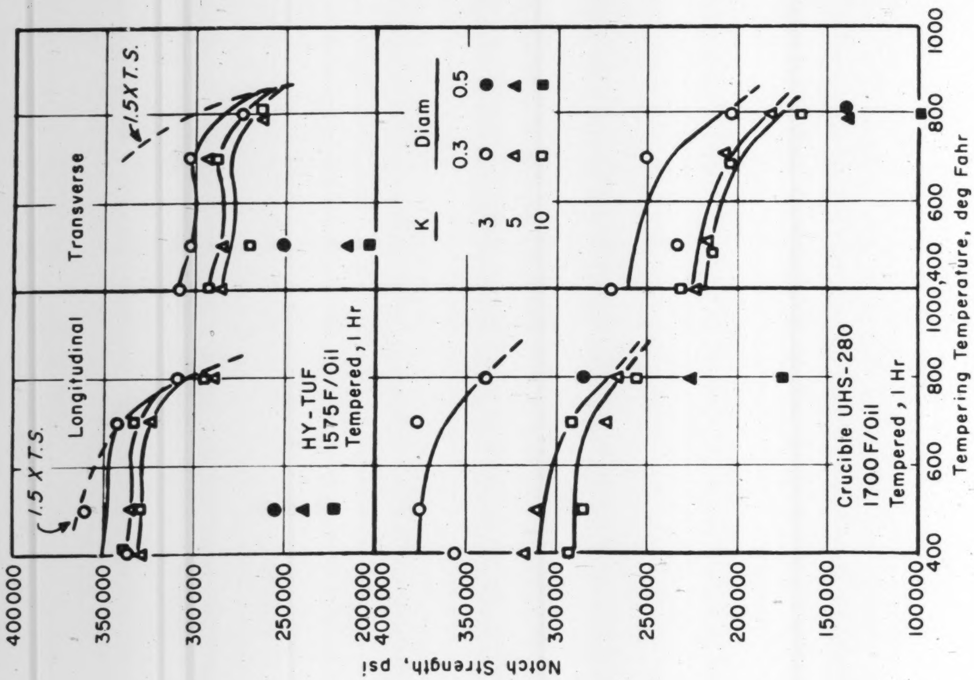

FIG. 13.—Notch Strength of Hy-Tuf and Crucible UHS-280 as a Function of Tempering Temperature.

*Notch Strengths:*

Of the steels examined, several, as it has already been stated, can be tempered to different tensile strengths while the remaining steels best provide for strengths within a specified range. On the other hand, if the tempering operation is completed in a manner to avoid the phenomenon of "500 deg embrittlement," the versatility of those steels which can be tempered to several tensile strengths is reduced. Therefore, to determine the tempering temperatures which provide optimum notch strength for the respective steels, the notch strength was determined as a function of the tempering temperature over the range of 250 to 800 F. The notch strength data in the longitudinal and transverse directions for the various steels are presented in Figs. 11 to 14.

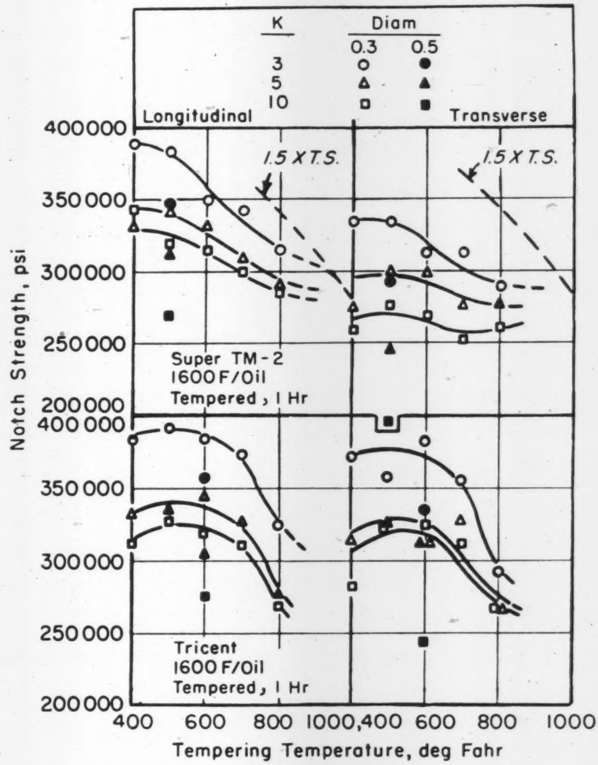

FIG. 14.—Notch Strength of Super TM-2 and Tricent as a Function of Tempering Temperature.

The trends established and discussed for 4340 steel are, in general, observed to exist in the case of the other steels in question. The notch strength of these steels was seen to depend upon the tempering temperature (strength level), stress concentration, specimen size, and specimen orientation. In general, the notch strength passed through a maximum and finally decreased with further increase in the tempering temperature. The notch strength was appreciably reduced as the stress concentration and specimen size were increased. The magnitude of these effects varied with the steel composition and was, in general, much more pronounced in the transverse than in the longitudinal direction. possible variation in properties in the two zones in accord with results reported by Bucknall (13).

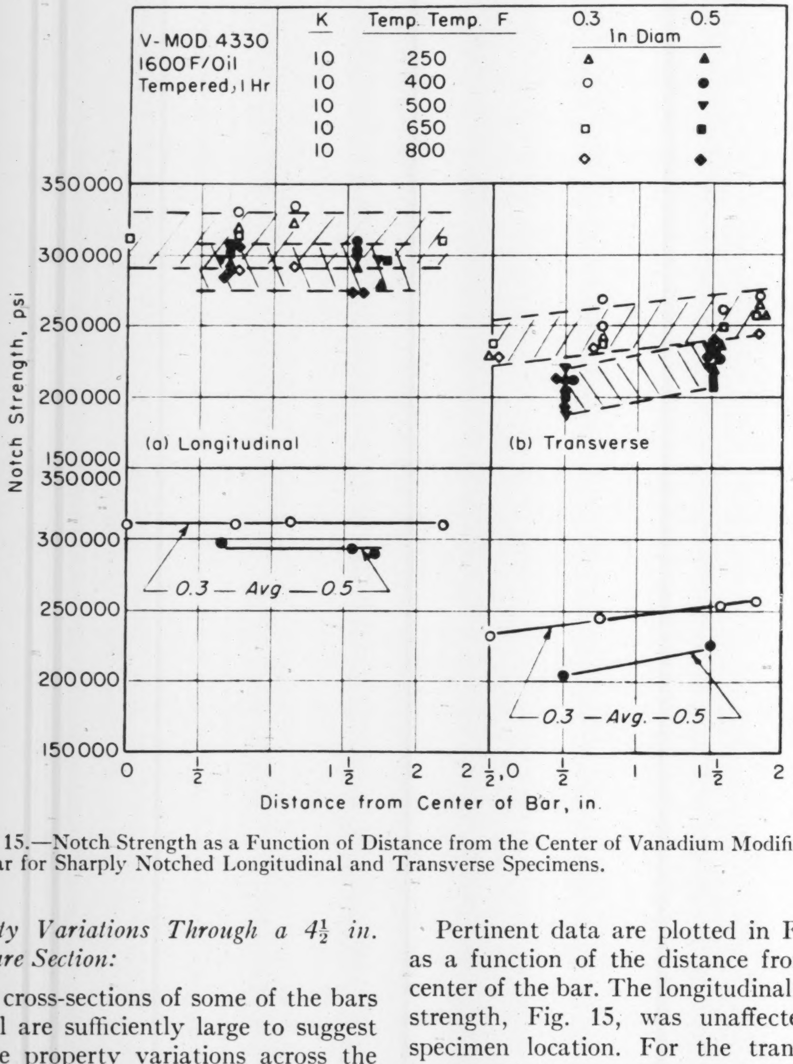

FIG. 15.—Notch Strength as a Function of Distance from the Center of Vanadium Modified 4330 Steel Bar for Sharply Notched Longitudinal and Transverse Specimens.

### *Property Variations Through a $4\frac{1}{2}$ in. Square Section:*

The cross-sections of some of the bars of steel are sufficiently large to suggest possible property variations across the section and in particular from the center to the surface of the bar. Further, the cross-section of the $4\frac{1}{2}$-in. bar of vanadium-modified 4330 steel, on macroetching, was observed to be divided into rim and core sections. This suggested a possible variation in properties in the two zones in accord with results reported by Bucknall (13).

Pertinent data are plotted in Fig. 15 as a function of the distance from the center of the bar. The longitudinal notch strength, Fig. 15, was unaffected by specimen location. For the transverse specimens, the notch strength, Fig. 15 (*b*), varied by less than 10 per cent as the specimen location was changed. A comparable analysis for the other steels indicated an even smaller variation.

## SUMMARY

The results presented in this paper lead to a number of conclusions which may be summarized as follows:

1. The various heats of 4340 steel studied in this program yielded, in general, comparable tensile and notch-tensile properties. The slight differences encountered in the transverse direction were observed to bear no correlation to differences in the hardenability of the various heats.

2. For all practical purposes, the various sizes of 4340 steel sections were observed to possess identical tensile and notch-tensile properties in the longitudinal direction. The transverse notch strength and ductility of the small sections, however, were appreciably lower than the respective values for the large sections.

3. An increase in the as-tested section size resulted in an appreciable decrease in the notch strength. This effect varied greatly with the steel composition.

4. The notch strength of high-strength steel specimens decreased with increase in the stress concentration. The magnitude of this effect, however, depended on various factors such as tempering temperature, loading direction and specimen size.

5. Eccentric loading resulted in an appreciable decrease in the notch strength of high-strength 4340 steel. An eccentricity of approximately 0.106 in. was observed to decrease the notch strength by about 60 ± 5 per cent.

6. Hydrogen contents considerably higher than those present in the steels here studied are necessary for developing a tendency to sustained-load failures.

7. The position of the specimen in the large sections investigated had a slight effect only on the transverse notch strength, which increased as the specimen location moved from the center to the rim of the section.

*Acknowledgments:*

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## REFERENCES

(1) G. Sachs, "Survey of Low-Alloy Aircraft Steels Heat-Treated to High-Strength Levels," Wright Air Development Center *Technical Report* 53–254, Part 3 (1954).

(2) G. Sachs, "Survey of Low-Alloy Aircraft Steels Heat-Treated to High-Strength Levels," Wright Air Development Center *Technical Report* 53–254, Part 4 (1953).

(3) G. Sachs and E. P. Klier, "Survey of Low-Alloy Aircraft Steels Heat Treated to High-Strength Levels," Wright Air Development Center *Technical Report* 53–254, Part 5 (1954).

(4) R. E. Peterson, *Stress Concentration Design Factors*, John Wiley & Sons, Inc., New York (1953).

(5) G. Sachs, B. B. Muvdi, and E. P. Klier, "Effect of a Number of Variables on the Mechanical Properties of Aircraft High-Strength Steels," Wright Air Development Center *Technical Report* 55–103 (1955).

(6) G. Sachs, V. Weiss, and E. P. Klier, "Effects of a Number of Heat-Treating and Testing Variables on the Notch Strength of 4340 Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 555 (1956).

(7) H. Kiessler and W. Connert, "Notched-Bar Tensile Tests with Super-Imposed Bending Strain," *Archiv. Eiesnhuettenwesen*, Vol. 14, p. 555 (1940, 1941).

(8) G. Sachs and J. D. Lubahn, "Bursting Tests on Notched Alloy Steel Tubing," *Transac-*

*tions*, Am. Soc. Metals, Vol. 32, p. 71 (1943).

(9) R. H. Raring and J. A. Rinebolt, "Static Fatigue of High-Strength Steel," *Transactions*, Am. Soc. Metals, Vol. 48, p. 198 (1956).

(10) R. P. Frohmberg, W. J. Barnett, and A. R. Troiano, "Delayed Failure and Hydrogen Embrittlement in Steel," Wright Air Development *Technical Report* 54–320 (1954).

(11) C. M. Carmen and D. F. Arimento, "Effect of Specimen Size on Hardenability," *Frankford Arsenal Report No. R-1164*, (Sept. 1954).

(12) J. Vajda and P. E. Busby, "Effect of Composition on Transverse Properties of Slack-Quenched Steel," *Transactions*, Am. Soc. Metals, Vol. 47, p. 408 (1955).

(13) E. H. Bucknall, "A Note on the Effect of the Location of the Test-Piece on the Jominy Hardenability of Billets," Iron and Steel Inst., *Special Rept. No. 36*, p. 120 (1946).

## DISCUSSION

MESSRS. FRANK A. MCCLINTOCK[1] AND MYER P. KUTZ.[2]—As we understand it, the stresses shown in Fig. 6 were obtained by dividing the load by the net area at the root of the notch in all cases. This practice of calculating stresses from nominal, or at best elastic stress distributions has been used often because of the lack of knowledge of plastic stress distributions, especially in the presence of notches. But in the case of eccentric loading, even an approximate plastic analysis may be of help in interpreting the results.

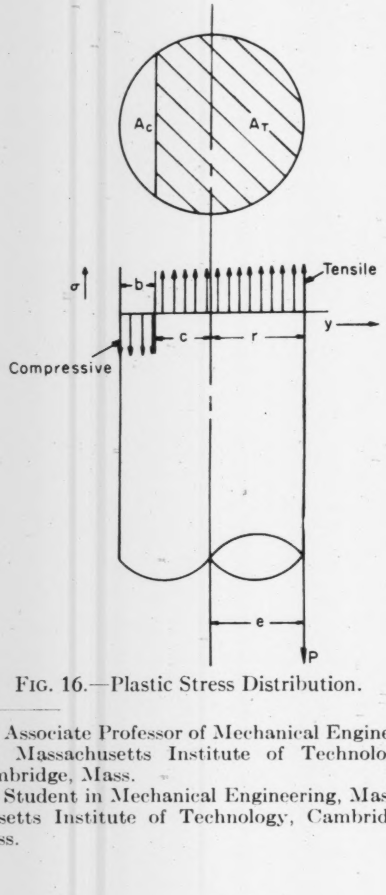

FIG. 16.—Plastic Stress Distribution.

Consider the distribution of stress in a smooth bar of non-workhardening material, subject to eccentric loading as shown in the accompanying Fig. 16. For sections in the middle of a long bar, symmetry arguments show that strain varies linearly across the section in the plastic as well as in the elastic case. Thus the plot of stress across the section is similar to the stress-strain curve. The plot may be approximated by constant tensile or compressive stress on two parts of the cross-sectional area.

For axial equilibrium, $\sigma_Y(A_T - A_c) = P$.

For rotational equilibrium, $\sigma_Y(\int_{A_T} Y dA - \int_{A_c} Y dA) = Pe$. From these relationships,

$$\sigma_y(2c\sqrt{r^2 - c^2} + 2r^2 \sin^{-1}(c/r)) = P$$

$$\sigma_y(4/3(r^2 - c^2)^{3/2} = Pe$$

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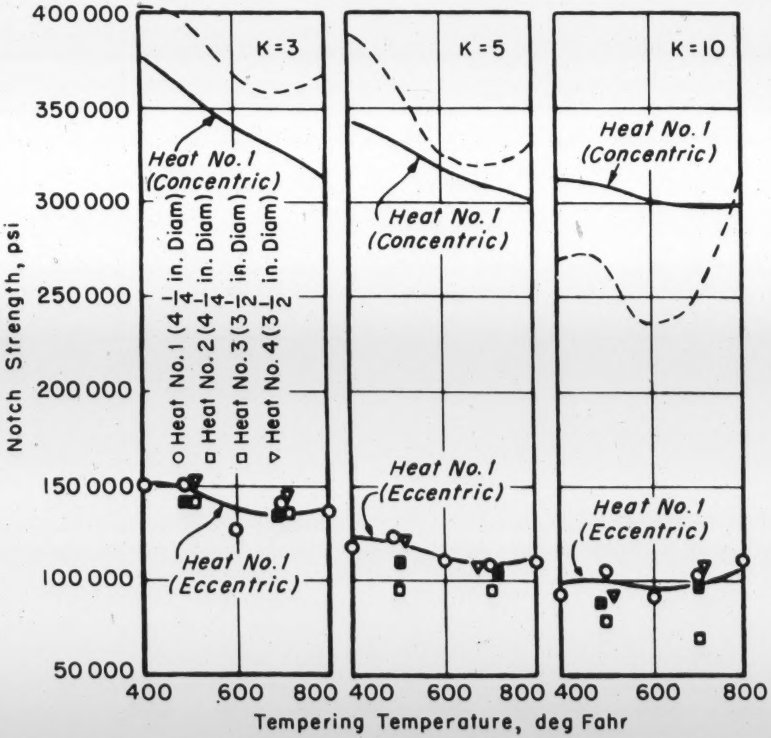

FIG. 17.—Eccentric Notch Strength According to Plastic Theory (Dashed lines).

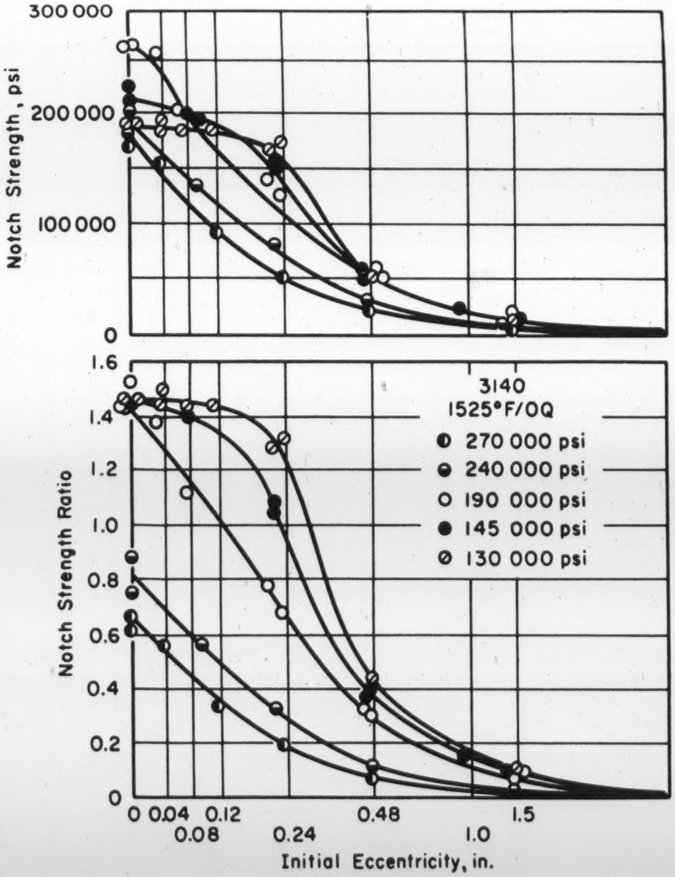

FIG. 18.—Effect of Eccentricity on Notch-Strength and Notch-Strength Ratio of Sharply Notched 3140 Steel Specimens Heat Treated to Various Strength Levels.

Trial and error solution of the above equations gives $c = 0.295\ r$ and a ratio of yield stress to nominal stress of $\sigma_Y/(P/\pi r^2) = 2.67$.

A corresponding elastic analysis, on the other hand, gives a ratio of maximum elastic stress to nominal stress of 5 to 1.

Taking the plastic case, and assuming that the increase in stress due to eccentricity is the same in a notched as in a smooth bar, and recalculating the effect of eccentricity accordingly from Fig. 6, one finds the results shown by the dashed line in Fig. 17. Apparently, then, the effect of eccentric loading is principally to increase the stress applied to the material locally under a given load rather than to reduce its strength. In other words, the problem seems to be more in the sphere of the stress analyst rather than the metallurgist. It is hoped that more cases will arise in the future where the specimens, the materials engineer tests have shapes the stress analyst can study, even in the presence of plastic deformation.

MR. E. P. KLIER (*author*).—It has been generally understood that the major reduction in the nominal notch strength measured under eccentric loading arises due to changes in the loading system. This reduction is a first order effect, and for this reason the application of an approximate plasticity theory leads to reasonable agreement between comparable sets of data as Messrs. McClintock and Kutz have shown. However, the element of notch sensitivity is a second order effect, and any usable theory must encompass second-order phenomena. For example, we can be quite certain that the analysis presented in the discussion will not apply if the specimen size is changed significantly.

At the present time the assertion that eccentric loading accentuates notch sensitivity in certain materials is based on experimental data such as presented in the accompanying Fig. 18.[3] Here the notch strength is measured as function of the eccentricity of loading. According to the plasticity theory advanced in the discussion these curves should form an homologous set and then by the introduction of suitable generalized coordinates all data could be represented by one curve. We are not persuaded that the data conform to this requirement, and the deviation from it is the element which we have attributed to notch sensitivity.

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[3] G. Sachs, J. D. Lubahn, and E. J. Ebert, "Notched Bar Tensile Test Characteristics of Heat-Treated Low-Alloy Steels," *Transactions*, Am. Soc. Metals, Vol. 33, p. 340 (1944).

# PROPERTIES OF OPEN-HEARTH AND BESSEMER SEAMLESS PIPE*

By A. B. Wilder[1] and W. P. Benter[1]

## Synopsis

This paper discusses certain properties of carbon steel pipe. Capped (rimmed) steels are customarily used for welded pipe, but in this instance were rolled into seamless pipe for comparison with the killed steels generally employed in the manufacture of seamless pipe. All the material tested was made into seamless pipe of the same size. Steels made by the basic open-hearth and acid bessemer processes were investigated.

The aging characteristics after cold working and also after quenching were evaluated and the various steels compared. The fatigue properties, impact properties and elevated temperature tensile properties were also investigated. Underhead cracking and girth-weld tests were made. The cracking susceptibility, macrostructure, microstructure, tension and bend tests and hardness properties of the welds were investigated.

Grade B bessemer seamless pipe had mechanical properties and welding characteristics similar to the grade B open-hearth seamless pipe. The toughness properties of the killed bessemer steels were similar to the open-hearth steels and were superior to the capped steels. The superior properties of deoxidized acid bessemer steel as compared with regular acid bessemer steel are shown.

In 1937 when the manufacture of lap-welded pipe was discontinued by the National Tube Division, United States Steel Corp., a new type of seamless pipe (1)[2] was developed. This material, which has now been manufactured for a period of 20 years, is made from deoxidized acid bessemer steel and has been included in ASTM specification A 53[3] since 1943. Deoxidized acid bessemer seamless pipe is a product which at the present time has a long and satisfactory record. It is recognized not only in ASTM specifications A 53 and A 120,[4] but is also included in the American Petroleum Inst. Standards 5A, 5L, and 5LX. The American Standards Association Code for Pressure Piping and The American Society for Mechanical Engineers Boiler and Pressure Vessel Code Committee recognize this as grade B bessemer seamless pipe.

The purpose of this investigation is to summarize the recent results obtained in a comparison of open-hearth and bessemer steels made with several different deoxidation practices and rolled into seamless pipe. Although capped (rimmed) open-hearth and bessemer steels in this investigation were rolled into seamless pipe for purposes of com-

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 749.

[3] Specification for Welded and Seamless Steel Pipe (A 53 – 55 T), 1955 Book of ASTM Standards, Part 1, p. 1.

[4] Specification for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 54, 1955 Book of ASTM Standards, Part 1, p. 47.

TABLE I.—CHEMICAL ANALYSIS AND TENSION PROPERTIES.

| Material[a] | Heats Tested | Composition, per cent | | | | | Carburized Austenitic Grain Size | Yield Strength, psi[b] | Ultimate Tensile Strength, psi | Elongation in 2 in., per cent |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Carbon | Manganese | Phosphorus | Sulfur | Silicon | | | | |
| Capped OH | 2 | 0.05 | 0.34 | 0.007 to 0.008 | 0.031 to 0.043 | 0.003 to 0.005 | 3 to 6 | 35 000 | 47 800 | 53 |
| Capped bess | 4 | 0.07 to 0.08 | 0.35 to 0.44 | 0.07 to 0.09 | 0.017 to 0.035 | 0.004 to 0.007 | 1 to 5 | 49 000 | 66 000 | 41 |
| Grade A OH-Al-Si | 3 | 0.13 to 0.20 | 0.44 to 0.62 | 0.009 to 0.023 | 0.022 to 0.024 | 0.15 to 0.20 | 7 to 8 | 37 400 | 64 500 | 43 |
| Grade B OH-Al-Ti | 3 | 0.25 to 0.28 | 0.55 to 0.63 | 0.009 to 0.016 | 0.038 to 0.042 | 0.027 to 0.078 | 6 to 8 | 36 900 | 67 700 | 42 |
| Grade B OH-Al-Si | 3 | 0.24 to 0.26 | 0.80 to 0.83 | 0.016 to 0.018 | 0.030 to 0.035 | 0.14 to 0.17 | 6 to 7 | 47 900 | 80 000 | 39 |
| Grade B OH-Si | 3 | 0.25 to 0.28 | 0.71 to 0.87 | 0.018 | 0.028 to 0.033 | 0.16 to 0.19 | 1 to 3 | 46 100 | 80 100 | 39 |
| Grade B bess-Al | 3 | 0.13 to 0.15 | 0.44 to 0.50 | 0.08 to 0.09 | 0.025 to 0.030 | 0.031 to 0.037 | 8 | 41 300 | 67 300 | 43 |
| Grade B bess Al-Si | 6 | 0.13 to 0.16 | 0.42 to 0.51 | 0.07 to 0.08 | 0.020 to 0.023 | 0.19 to 0.22 | 6 to 8 | 45 500 | 71 900 | 42 |

[a] OH—Open-hearth, bess—bessemer.

[b] Drop of beam method.

parison, this practice, for economic reasons, is not used for the production of seamless pipe. Capped bessemer steel was first used for the production of butt-welded pipe in 1887 by National Tube and has been successfully used for this purpose up to and including the present time.

Previous investigations of open-hearth and bessemer seamless pipe were directed toward evaluation of the toughness (2) and weldability (3) characteristics of the material. Some of these properties and others, including aging, high-temperature tensile strength and fatigue-endurance limits will be discussed with reference to the present investigation.

## STEEL MAKING PRACTICE

Steel made by the tilting and stationary basic open-hearth processes (4) and the acid bessemer process (5) were employed in this investigation. Although both capped and killed steels were rolled into seamless pipe, capped steels have been rarely used to produce seamless pipe due to the lack of uniform practice.

Deoxidized or killed acid bessemer steel (5) used in this investigation is not only deoxidized in the ladle with silicon and aluminum, but also with carbon from molten pig iron as an addition to the converter at the end of the blow. In this special practice, a considerable portion of oxygen is removed as carbon-monoxide gas by the addition of molten pig iron, producing a clean steel. Deoxidized-acid bessemer steel used for the manufacture of seamless pipe is always killed and should, therefore, be distinguished from ordinary bessemer steel, which is capped or rimmed. Too frequently this distinction is not made, with the result that considerable confusion has existed in distinguishing between the two types of steel.

## MATERIALS

The materials used in this investigation were hot-rolled seamless pipe $6\frac{5}{8}$ in. in outside diameter with a 0.562 in. wall made in accordance with applicable parts of ASTM specification A 53. Various types of steel were studied and the grade A and grade B materials were typical for these materials in ASTM specification A 53. Open-hearth and bessemer-capped steels commonly used in the production of furnace butt-welded pipe were rolled into seamless pipe so that steels with suitable size and processing history would be available for comparison with killed seamless steels. In addition to the capped steels, open-hearth steel made with aluminum-silicon deoxidation practice was rolled into grade A seamless pipe, and with silicon, aluminum-silicon and aluminum-titanium deoxidation practice were rolled into grade B seamless pipe. Bessemer steel made with aluminum-silicon and aluminum deoxidation practice was rolled into grade B seamless pipe. Only a small amount of seamless pipe is produced from semi-killed steel (2), and therefore this type of deoxidation practice was not included in the current investigation.

The chemical analysis, tension properties and ASTM austenitic grain size[5] after carburizing the steels are shown in Table I. The residual alloy content of these steels was very low, as no steel scrap containing alloys was used in steel melting. The grade B bessemer seamless has a higher phosphorus content resulting in the advantage of a lower carbon content to produce the required mechanical properties. All the steels had a fine austenitic grain size after carburizing for 8 hr at 1700 F, with the exception of the capped steels and the silicon-killed grade B open-hearth steel.

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[5] Classifications of Austenitic Grain Size in Steels (E 19 - 46 T), 1955 Book of ASTM Standards, Part I, p. 1602.

All material was tested in the as-rolled condition with the exception of the material which was quench-aged. The results of hardenability tests of end-quenched 0.5-in. diameter solid bars, made in accordance with ASTM Method A 255,[6] are shown in Fig. 1. Hardenability of the killed or deoxidized steels was greater when tested from 2100 F as compared with 1600 F. The increased hardenability was due to the larger grain size. The hardenability of the grade B bessemer steel was less than that of the open-hearth steels. Hardenability of the capped steels was at a very low level due to the low carbon content of these steels.

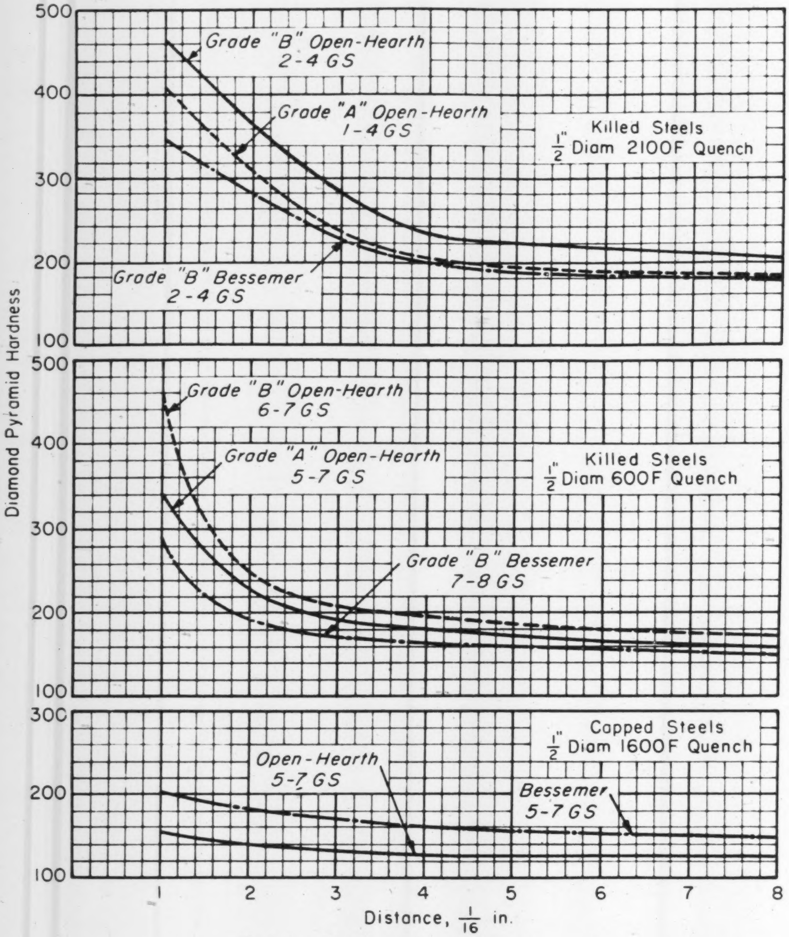

FIG. 1.—Hardenability of Steels.

All of the material subjected to the standard flattening test was satisfactory. This test consisted of flattening a $2\frac{1}{2}$-in. long pipe ring in compression in accordance with the grade in ASTM specification A 53. Macroscopic etch tests of pipe rings indicated satisfactory soundness for the killed steels. In the deep etch tests the open-hearth capped steels exhibited a rimmed area of approximately 50 per cent of the wall thickness. In the bessemer capped steel there was no core and rim, but lack of soundness in the etched area was observed, which is characteristic of capped steels.

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[6] Tentative Method of End-Quench Test for Hardenability of Steel (A 255 – 48 T), 1955 Book of ASTM Standards, Part 1, p. 636.

## TEST METHODS

### *Aging Tests:*

The aging characteristics of the steel were evaluated with a 0.450-in. in diameter, round Izod impact test specimen (2). The specimen had a 45-deg. notch, 0.13 in. in depth with a radius of 0.01 in. The coldworked specimen was prepared from a round tapered bar (2). The specimens were tested in a Charpy-Izod impact testing machine, loaded as a cantilever beam.

### *Fatigue Tests:*

A standard 0.300-in. diameter polished fatigue specimen and a similar specimen with a notch 0.0050 in. in depth having a radius of 0.0062 in. were used. These specimens were two-point loaded in an R. R. Moore fatigue testing machine and tested at 1790 rpm. The fatigue strength was based on the maximum load for ten million cycles without failure. Eight to twelve samples were employed to establish the fatigue strength.

### *Impact Tests:*

Impact test specimens other than those employed in the aging tests were standard full-size Charpy keyhole specimens. Longitudinal samples were machined as depicted in a previous investigation (2). The test specimens were loaded as a simple beam and tested in a temperature range of −20 F to 750 F in accordance with the ASME Boiler and Pressure Vessel Construction Code, Paragraph G - 1.[7]

Tests were made with a notch in the unaffected base material and in the heat-affected zone of the girth weld. It was recognized that some uncertainty exists in determining the impact properties of the heat-affected zone. The impact specimens with the heat-affected zone were notched from the root side of the weld and fracture occurred through the fine- and coarse-grained material.

### *Tension Tests:*

Tension test results in Table I are based upon standard ASTM strip-test specimens[8] which were 1.5-in. wide and 0.56 in. in thickness with a gage length of 2 in. Tension tests of girth welds were made in accordance with the ASME Boiler and Pressure Vessel Construction Code.[9] A reduced section tension specimen $\frac{3}{4}$-in. wide was also employed.

### *Girth Welds:*

Circumferential manual and automatic metallic arc welds of full pipe sections were used in this investigation. The welds were made with as-rolled pipe by a pipe fabricator in accordance with the ASME Boiler and Pressure Vessel Construction Code.[9] The silicon and aluminum-silicon grade B open-hearth seamless and aluminum-silicon grade B bessemer seamless pipe samples were scribed (2). This procedure utilized an E-6010 electrode and a 37½ deg bevel and was accomplished in five welding passes. All other pipe samples were automatic welded with five passes, a Lincoln L-60 electrode with L-760 flux and a 30 deg bevel being used. Preheat, postheat, or stress relieving were not used during welding. The pipe was rolled during welding and therefore welded in a flat position. The composite welds of bessemer are open hearth steels and grades A and B open hearth steels were studied because these conditions exist in piping systems. It is therefore desirable to evaluate the effect of various chemical compositions when they are joined with weld metal.

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[7] G-1 Rules for Construction of Unfired Pressure Vessels, Section VIII, American Society of Mechanical Engineers, Boiler and Pressure Vessel Construction Code (1956).

[8] Tentative Methods and Definitions for Mechanical Testing of Steel Products (A 370 – 54 T), 1955 Book of ASTM Standards, Part 1, p. 351.

[9] Qualification Standard for Welding Procedures and Welding Operators, Section IX, American Society of Mechanical Engineers, Boiler and Pressure Vessel Construction Code (1956).

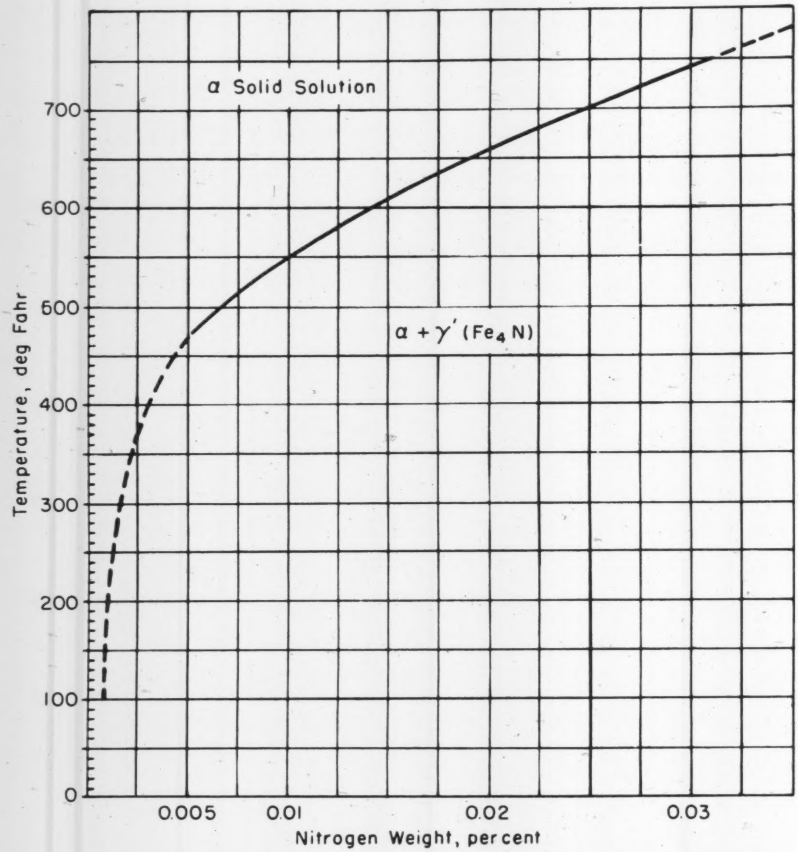

FIG. 2.—Solubility of Nitrogen in Iron After Rawlings and Tambini (7).

Guided- and free-bend tests were used for evaluating soundness and ductility of manually welded, which has been de- the girth welds. Three tests were made in accordance with the ASME Boiler and Pressure Vessel Construction Code. The free-bend-test specimens were 0.5-in. thick and 0.75-in. wide. The root- and face-guided bend test specimens were 0.562-in. thick and 1½-in. wide.

*Underbead Cracking Tests:*

A weld bead (2) was deposited in a flat position at 0 F using a $\frac{1}{8}$-in. E-6010 electrode with 100 amp at 10 in. per min travel speed. The longitudinal weld bead $1\frac{1}{4}$-in. long was deposited on three different segments from a 3-in. long ring of pipe. After welding, the specimens were heated to 1100 F for 1 hr and longitudinally sectioned through the weld. The vertical plane through the weld was ground, polished, and etched to locate the heat-affected zone. The etched specimens were magnetic particle tested and the length of cracks were measured in percentage of total weld length cracked.

*Hardness Surveys:*

Hardness surveys were made on transverse sections of the girth (circumferential) and bead welds (2). A standard diamond pyramid hardness tester was used with 1- and 10-kg loads. The 1-kg load was used in the heat-affected zone. The macrostructure and microstructure of transverse sections of the girth and bead welds were observed.

## RESULTS AND DISCUSSION

*Aging:*

There are two general types of aging which were studied in this investigation: (1) aging after cold work, and (2) aging after quenching. Of the two types, aging after cold work is the most important with respect to tubular products. In the present study, steels, after cold working, were artificially aged at 450 F for 1 hr, and steels, after quenching in brine at 20 F from 1250 F and also 1700 F, were artificially aged at 212 F for 24 hr. The two methods of aging are not equivalent with each other. There are many methods for evaluating aging, but the most common property to evaluate aging in tubular products is by the change in toughness.

Many theories have been advanced to

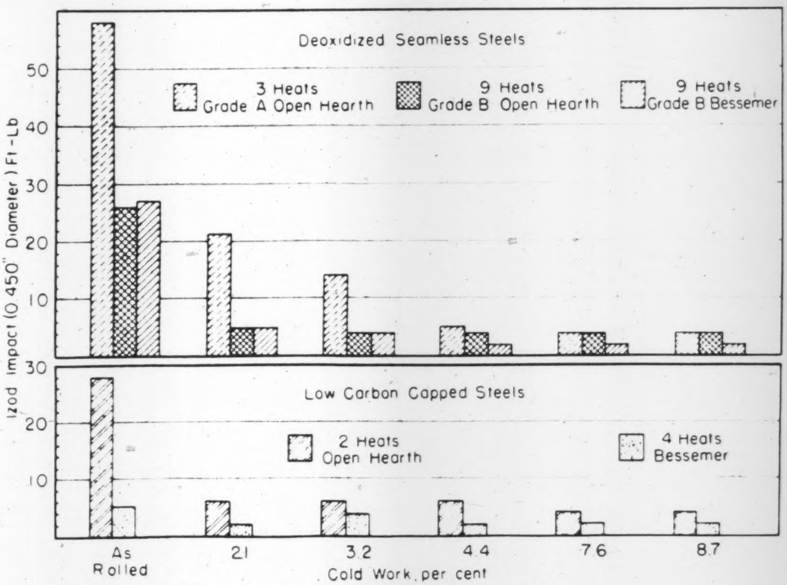

FIG. 3.—Impact Properties of Cold-Worked Steel.

explain the aging phenomena in steel. Cottrell (6) and his co-workers have developed a theory which involves crystal dislocations, bound in place by "atmospheres" of carbon or nitrogen atoms. It is, therefore, important to consider the carbon and nitrogen content of steel and the condition in which the elements exist. In order for steel to be susceptible to aging, it is essential that carbon or nitrogen be in solid solution. The solubility of nitrogen in steel is shown in Fig. 2, and a similar type of curve may be depicted for carbon in steel. It will be observed from the solubility curve in Fig. 2 that supersaturation of $Fe_4N$ in alpha iron may be achieved by cooling rapidly from higher temperatures in the presence of sufficient nitrogen to exceed the solubility of $Fe_4N$ in alpha iron at the lower temperature. Supersaturation at ambient temperature may also be achieved by cold work which, after aging, results in dislocations.

TABLE II.—QUENCH AGING RESULTS.

| Material | Heats Tested | As-Rolled | | Quenched 1250 F, Aged 212 F | | Quenched 1700 F, Aged 212 F | |
|---|---|---|---|---|---|---|---|
| | | Impact,[a] ft-lb | Rockwell Hardness, B scale[a] | Impact, ft-lb | Rockwell Hardness, B scale | Impact, ft-lb | Rockwell Hardness, C scale |
| Capped open-hearth | 2 | 28 | 51 | 7 | 84 | 79 | 7[b] |
| Capped bessemer | 4 | 5 | 75 | 3 | 90 | 38 | 34 |
| Grade A open-hearth | 3 | 58 | 69 | 19 | 88 | 27 | 41 |
| Grade B open-hearth | 9 | 26 | 81 | 19 | 91 | 13 | 48 |
| Grade B bessemer | 9 | 27 | 78 | 23 | 90 | 24 | 40 |

[a] 0.450 in. diameter notched izod impact test—Rockwell hardness B scale.

[b] 88 Rockwell B equivalent to 7 Rockwell C.

In Fig. 2, we have the alpha iron and $Fe_4N$ constituents. In the manufacture of deoxidized acid bessemer seamless steel, aluminum is added for nitrogen fixation. The total nitrogen content of deoxidized acid bessemer (seamless) steel therefore does not indicate the amount of $Fe_4N$ present, as a certain amount of nitrogen has combined with aluminum to form aluminum nitride. In the aging phenomena we are concerned with carbon as $Fe_3C$ and nitrogen as $Fe_4N$. Therefore, the fixation of nitrogen with aluminum in killed bessemer (seamless) steel is responsible for the fact that the aging characteristics are similar to open-hearth steel. Further, if bessemer or open-hearth steel properly killed with aluminum is normalized, the aging susceptibility characteristics after cold working are appreciably reduced. This is due to the further fixation of nitrogen as aluminum nitride by heat treatment. The fixation of nitrogen explains in part the beneficial results of normalizing and the behavior of hot-rolled steel properly killed with aluminum.

Work-brittleness test results are shown in Fig. 3. Grade A material when cold worked a small amount, had the higher impact strength when compared with grade B steels. After additional cold deformation, the various deoxidized steels used for regular production of seamless pipe were similar in their behavior. Work-brittleness tests of the capped steels not used for regular production of seamless pipe indicated brittleness with small amounts of cold work. The work-brittleness test results show that cold working embrittles all the steels tested, and grade B bessemer seamless pipe is similar in behavior to grade B open-hearth seamless pipe.

TABLE III.—FATIGUE PROPERTIES.

| Material | Heats Tested | Endurance Limit, psi | | Ratio, Notch Fatigue / Un-Notched Fatigue × 100 | Ratio, Un-Notched Fatigue / Ultimate Tensile × 100 | Ratio, Notched Fatigue / Ultimate Strength × 100 |
|---|---|---|---|---|---|---|
| | | Un-Notched | Notched | | | |
| Capped open-hearth | 2 | 27 000 | 20 500 | 76 | 58 | 41 |
| Capped bessemer | 4 | 38 000 | 27 000 | 71 | 59 | 35 |
| Grade A open-hearth aluminum-silicon | 3 | 33 000 | 25 300 | 77 | 53 | 40 |
| Grade B open-hearth aluminum-titanium | 3 | 28 700 | 22 500 | 78 | 44 | 35 |
| Grade B open-hearth aluminum-silicon | 3 | 38 700 | 27 300 | 71 | 46 | 32 |
| Grade B open-hearth silicon | 3 | 37 300 | 27 000 | 72 | 45 | 33 |
| Grade B bessemer aluminum | 3 | 38 300 | 27 000 | 71 | 59 | 42 |
| Grade B bessemer aluminum-silicon | 6 | 43 100 | 28 100 | 65 | 56 | 37 |

TABLE IV.—CHARPY KEYHOLE NOTCH IMPACT TESTS OF GIRTH WELDS AVERAGE VALUES, FT-LB.

| Testing Temperature, deg Fahr | Unaffected Base Metal | | | | | Heat-Affected Zone | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Capped[a] | | Killed[a] | | | Capped | | Killed | | |
| | OH | B | OH Grade A | OH Grade B | B Grade B | OH | B | OH Grade A | OH Grade B | B Grade B |
| | 1 Heat | 4 Heats | 3 Heats | 9 Heats | 9 Heats | 1 Heat | 3 Heats | 3 Heats | 9 Heats | 9 Heats |
| −20 | 3 | 1 | 18 | 8 | 16 | 5 | 3 | 47 | 37 | 37 |
| 0 | 5 | 2 | 30 | 14 | 18 | 26 | 4 | 48 | 43 | 39 |
| 25 | 6 | 2 | 32 | 25 | 25 | 64 | 32 | 62 | 54 | 46 |
| 75 | 17 | 2 | 41 | 33 | 32 | 83 | 38 | 66 | 49 | 51 |
| 450 | 81 | 38 | 54 | 36 | 45 | 86 | 42 | 59 | 45 | 47 |
| 750 | 88 | 29 | 33 | 23 | 27 | 36 | 30 | 42 | 27 | 28 |

[a] OH—Open-hearth, B—bessemer.

The quench aging results in Table II show the capped steels after quenching from 1700 F and aging was superior to the killed grade A and B steels. However, when quenched from 1250 F, the killed steels were superior to the capped steels. Results obtained with killed bessemer seamless pipe indicated this material was similar to killed open-hearth seamless steel pipe, and neither was embrittled by quench aging.

*Behavior in Fatigue:*

The fatigue properties of the capped and killed steels are summarized in Table III. The fatigue strength of the capped bessemer steel was superior to the capped open-hearth steel. This was due to the higher strength level of the capped bessemer steel, which was due to the phosphorus content of this material.

The grade B killed steels were similar, with the exception of the aluminum-titanium deoxidized open-hearth steel, which had lower fatigue properties. The notch fatigue properties of the killed steels were essentially similar. The grade B killed bessemer steel in notched and unnotched fatigue was similar to the grade B killed open-hearth steel. The bessemer seamless steel was no more notch sensitive in fatigue than the open hearth (seamless) steel.

The killed steels had a high level of impact properties in both the unaffected base material and weld heat-affected zone. The open-hearth grade B material had the lower impact strength at −20 F, but in the weld heat-affected zone the impact strength was high. The impact properties of all the steels were higher in the weld heat-affected zone compared with the original base material. At 750 F the impact properties were lower compared with 450 F, but the steels exhibited a high level of toughness. The impact properties of the grade B killed bessemer (seamless) steel were superior to the grade B killed open-hearth (seamless) steel.

TABLE V.—TENSION AND BEND TEST RESULTS OF CIRCUMFERENTIAL WELDS.

| Material | Heats Tested | Tension Tests | | | Bend Tests | | |
|---|---|---|---|---|---|---|---|
| | | Yield Strength, psi | Tensile Strength, psi | Elongation in 2 in., per cent | Free Bend Elongation, per cent | Guided Bend | |
| | | | | | | Face | Root |
| Capped open-hearth | 1 | 41 000 | 67 000 | 53 | 40 | OK | OK |
| Capped bessemer | 4 | 41 000 | 66 000 | 29 | 57 | OK | OK |
| Grade A open-hearth | 3 | 31 000 | 50 000 | 32 | 55 | OK | OK |
| Grade B open-hearth | 9 | 48 000 | 73 000 | 22 | 68 | OK | OK |
| Grade B bessemer | 9 | 46 000 | 72 000 | 27 | 59 | OK | OK |
| Grade A open-hearth welded to Grade B open-hearth | 3 | 40 000 | 66 000 | 29[a] | 68 | OK | OK |
| Grade A open-hearth welded to Grade B bessemer | 3 | 45 000 | 65 000 | 31[a] | 65 | OK | OK |
| Grade B open-hearth welded to Grade B bessemer | 3 | 45 000 | 68 000 | 30[b] | 70 | OK | OK |
| ASME Boiler code minimum requirements for grade B | | | 60 000 | | 25 | | |

[a] Fracture in grade A parent metal.

[b] Fracture in open-hearth parent metal in certain tests and bessemer parent metal in other tests.

*Impact Properties:*

The Charpy keyhole impact test results of the unaffected base material and the heat-affected zone of the girth welds in capped and killed steels are summarized in Table IV. The impact properties at temperatures below 75 F in the unaffected base plate material of the capped steels indicated a low level of toughness. In the weld heat-affected zone, the bessemer and open-hearth capped steels indicated a low level of toughness at −20 F. At 450 F and 750 F the impact properties indicated a high level of toughness in the girth welds of capped steels.

*Tension and Bend Test Results of Girth Welds:*

Tension properties and bend-test results are shown in Table V. All of the material met ASTM specification A 53 test requirements for pipe and the ASME Boiler and Pressure Vessel Construction Code test requirements for girth welding of pipe.

Of particular interest in this investiga-

## TABLE VI.—CIRCUMFERENTIAL WELD AND WELD BEAD DIAMOND PYRAMID HARDNESS NUMBER.

| Material[a] | Heats Tested | Girth Weld[b] | | | Longitudinal Bead Weld[b] | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Normal Welding Procedure | | | Severe Welding Procedure | | |
| | | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 |
| Capped OH | 2 | | | | 113 to 119<br>114 | 129 to 178<br>149 | 160 to 193<br>173 | 95 to 124<br>111 | 135 to 201<br>175 | 216 to 242<br>229 |
| Capped B | 4 | | | | 146 to 164<br>155 | 137 to 164<br>152 | 168 to 201<br>180 | 175 to 239<br>202 | 159 to 197<br>180 | 212 to 264<br>237 |
| Grade A-OH<br>Al-Si | 3 | 125 to 140<br>131 | 115 to 159<br>141 | 131 to 172<br>149 | 143 to 158<br>153 | 168 to 229<br>194 | 168 to 197<br>185 | 133 to 165<br>146 | 197 to 313<br>234 | 216 to 235<br>222 |
| Grade B-OH<br>Al-Ti | 3 | 131 to 145<br>138 | 131 to 168<br>148 | 135 to 169<br>148 | 135 to 173<br>155 | 165 to 229<br>193 | 176 to 193<br>180 | 128 to 155<br>142 | 224 to 313<br>268 | 228 to 251<br>243 |
| Grade B-OH<br>Al-Si | 3 | 146 to 171<br>158 | 145 to 219<br>172 | 134 to 170<br>150 | 155 to 180<br>170 | 224 to 276<br>252 | 179 to 198<br>189 | 159 to 175<br>164 | 330 to 401<br>363 | 248 to 285<br>272 |
| Grade B-OH<br>Si | 3 | 140 to 181<br>164 | 142 to 219<br>180 | 132 to 168<br>147 | 172 to 186<br>178 | 214 to 283<br>253 | 187 to 215<br>197 | 160 to 183<br>171 | 283 to 339<br>309 | 251 to 281<br>263 |
| Grade B-B<br>Al | 3 | 124 to 137<br>132 | 129 to 165<br>145 | 136 to 180<br>150 | | | | | | |
| Grade B-B<br>Al-Si | 6 | 138 to 168<br>152 | 143 to 197<br>171 | 133 to 167<br>148 | 148 to 178<br>160 | 193 to 239<br>221 | 191 to 225<br>204 | 147 to 165<br>157 | 210 to 263<br>234 | 262 to 287<br>271 |

[a] OH—Open-hearth, B—bessemer.
[b] 1—Unaffected pipe material, 2—weld heat-affected zone, 3—weld metal.

tion are the girth welds of dissimilar steels. Tests have been conducted in the field welding of hundreds of miles of line pipe in which grade B killed bessemer seamless pipe was welded to grade B killed open-hearth seamless pipe and no difficulty was experienced. The present tests were conducted to provide additional data on the strength and ductility properties of these welds. In addition, hardness tests and the microstructure were investigated and will be discussed later.

TABLE VII.—CIRCUMFERENTIAL WELD DIAMOND PYRAMID HARDNESS.

| Material | Heats Tested | Diamond Pyramid Hardness | | | | |
|---|---|---|---|---|---|---|
| | | Grade A Open-Hearth | | Weld Metal | Grade B Open-Hearth | |
| | | Unaffected base metal | Heat-affected zone | Weld metal | Heat-affected zone | Unaffected base metal |
| Grade A open-hearth aluminum-silicon welded to grade B open-hearth aluminum-titanium.......... | 3 | 125 to 140<br>131 | 115 to 162<br>141 | 131 to 172<br>149 | 110 to 201<br>146 | 129 to 161<br>139 |
| | | Grade A Open-Hearth | | Weld Metal | Grade B Bessemer | |
| Grade A open-hearth aluminum-silicon welded to grade B bessemer aluminum.................. | 3 | 133 to 163<br>139 | 135 to 175<br>153 | 136 to 180<br>150 | 129 to 165<br>145 | 124 to 137<br>132 |
| | | Grade B Open-Hearth | | Weld Metal | Grade B Bessemer | |
| Grade B open-hearth aluminum-titanium welded to grade B bessemer aluminum.................. | 3 | 131 to 145<br>138 | 131 to 168<br>148 | 135 to 169<br>148 | 131 to 197<br>152 | 133 to 160<br>144 |

In welding grade A killed open-hearth seamless pipe to grade B killed open-hearth or killed bessemer seamless pipe the strength of the joint generally depends upon the strength of the grade A material, provided the tension properties of the grade A material do not exceed the tension properties of the grade B material. In welding grade B killed bessemer seamless to grade B killed open-hearth seamless, the strength properties of the welded joint depend on the material with the lower strength. The type of steel is therefore not related to the strength properties of the girth weld.

In girth welding the capped steels, no difficulty was experienced with the rim and core sections of the weld, and satisfactory welds were obtained in this heavy wall material. The grade B killed bessemer (seamless) steel was welded without difficulty, and the tension and bend test properties of the girth welds were equivalent to the killed open-hearth steel.

### *Hardness Results of Girth and Bead Welds and Weld Bead Cracking Susceptibility:*

Diamond pyramid hardness results of the girth welds and bead welds are shown in Table VI. The hardness of the bead welds for capped steels was lower than the killed steels. Although the hardness of the capped bessemer steel exceeded the hardness of the capped open-hearth steels, the hardness of the heat-affected zones and weld metal were similar. The hardness results of the bead welds of killed grade B material were similar, with the exception that the hardness in the heat-affected zone of certain open-hearth heats exceeded the hardness of the other materials. This was due to the higher carbon content and different deoxidation practice for the open-hearth steels. In the severe bead welding procedure, hardness of the heat-affected zone exceeded the hardness of a similar zone in the normal bead welding procedure in this investigation.

In the girth weld hardness surveys, the tests were conducted on killed steels. Hardness results were similar for all the killed steels with exception of grade A open-hearth, grade B aluminum-titanium, and grade B aluminum-silicon steels. These latter three steels had a lower hardness level in the heat-affected zone and unaffected base material. In Table VII the hardness tests of girth welds with dissimilar steels are summarized. The results in general were similar for all the girth welds, including the welds of grade A open-hearth to grade B open-hearth, and grade B bessemer and grade B open-hearth to grade B bessemer.

TABLE VIII.—CRACKING SUSCEPTIBILITY TEST RESULTS.

| Material | Heats Tested | Percentage Cracked |
|---|---|---|
| Capped open-hearth | 3 | 0 |
| Capped bessemer | 4 | 0 |
| Grade A open-hearth | 3 | 0 |
| Grade B open-hearth | 9 | 28[a] |
| Grade B bessemer | 9 | 0 |

[a] Experience has indicated that, due to the severity of the test, cracking should exceed 50 per cent before preheating is desirable.

The cracking susceptibility test results summarized in Table VIII show that the grade B killed bessemer seamless pipe is similar to the grade A killed open-hearth seamless pipe and the capped steels. The only cracking encountered, which was in the grade B killed open-hearth material, was due to its higher carbon content. The grade B aluminum-titanium killed open-hearth steel did not crack. A level of 28 per cent cracking susceptibility is not considered objectionable, as the test is more severe than the conditions usually encountered in the field girth welding.

In the study of hardness of the girth and bead welds, carbon content of the original material was the most important factor. The grade B killed bessemer seamless pipe was superior to the grade B open-hearth seamless pipe in these tests. The lower carbon content of the killed bessemer seamless pipe contributed to the lower level of hardness encountered.

*Microstructure of Girth and Bead Welds:*

Typical metallographic structures of the girth welds of the capped steels are shown in Figs. 4 and 5. In the fusion zone of the root and filler beads and in the weld metal of root and filler beads, a fine grain structure was observed. This desirable structure was due to the effect of the weld heat which caused a recrystallization of the metal. The "as cast" weld metal and heat-affected zone in the cover bead were coarse-grained.

In the girth weld of grade A killed steel, the metallographic structures in Fig. 5 were similar to the capped steels, but at a higher carbon level. In Fig. 6 the metallographic structures obtained in girth welds of dissimilar grade A and grade B material are depicted. No unusual metallographic structures were observed in the grade A killed open-hearth seamless pipe welded to the grade B killed open-hearth or grade B killed bessemer steel pipe. Also, in welding grade B killed open-hearth to grade B killed bessemer seamless pipe, no unusual metallographic structures were observed.

The advantages of multipass welding are clearly shown by the grain refinement in the weld metal and heat-affected zone of the weld. The microstructure of girth welds of grade B killed open-hearth seamless pipe and grade B killed bessemer seamless pipe are described in an earlier investigation (**3**).

In the cracking susceptibility tests a severe welding procedure was employed, and these specimens were selected for metallographic examination. The capped steels in Figs. 7 and 8 did not show an acicular microstructure in the heat-affected zone, while this structure was observed in the grade A material, Fig. 8, and in the grade B killed steels. Typical microstructures of grade B killed open-hearth and killed bessemer seamless steels have been described in a previous investigation (**3**).

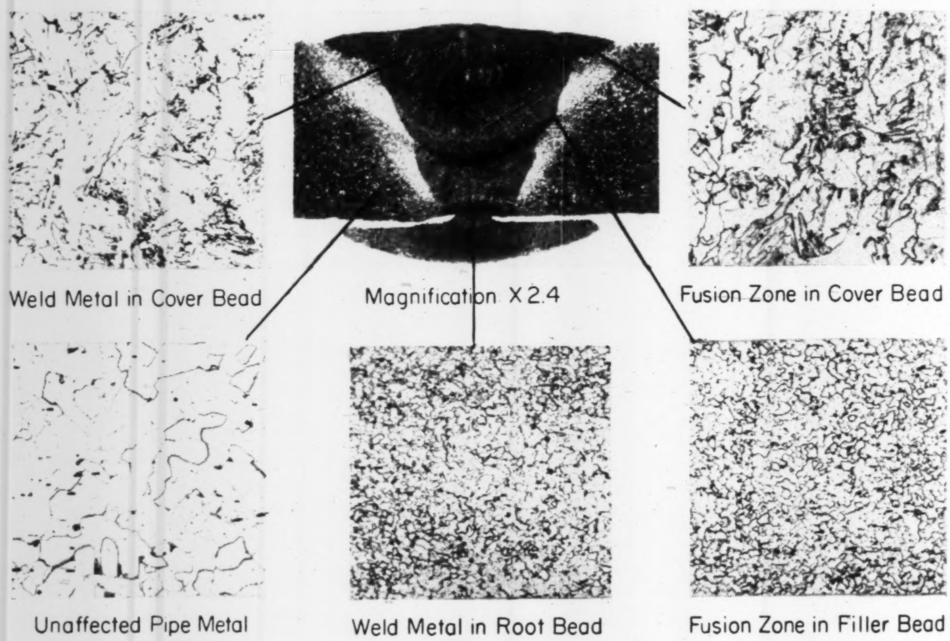

FIG. 4.—Microstructure of Girth Welds—Capped Open-Hearth Steel. Reduced to about $\frac{7}{10}$ size in reproduction.

In the capped steels, the width of the heat-affected zone in the girth welds is less than in the grade A or grade B killed steels. This may be explained on the basis of hardenability or chemical composition. The grade B killed bessemer seamless pipe had a microstructure comparable with grade B killed open-hearth seamless pipe. The higher phosphorus content of the grade B killed bessemer seamless pipe which permitted a lower level of carbon for grade B material resulted in more free ferrite in the killed bessemer steel microstructure.

## CONCLUSIONS

1. The grade B killed bessemer seamless pipe was equivalent to the grade B killed open-hearth seamless pipe with respect to tension, aging, fatigue, impact and welding properties of the materials tested.

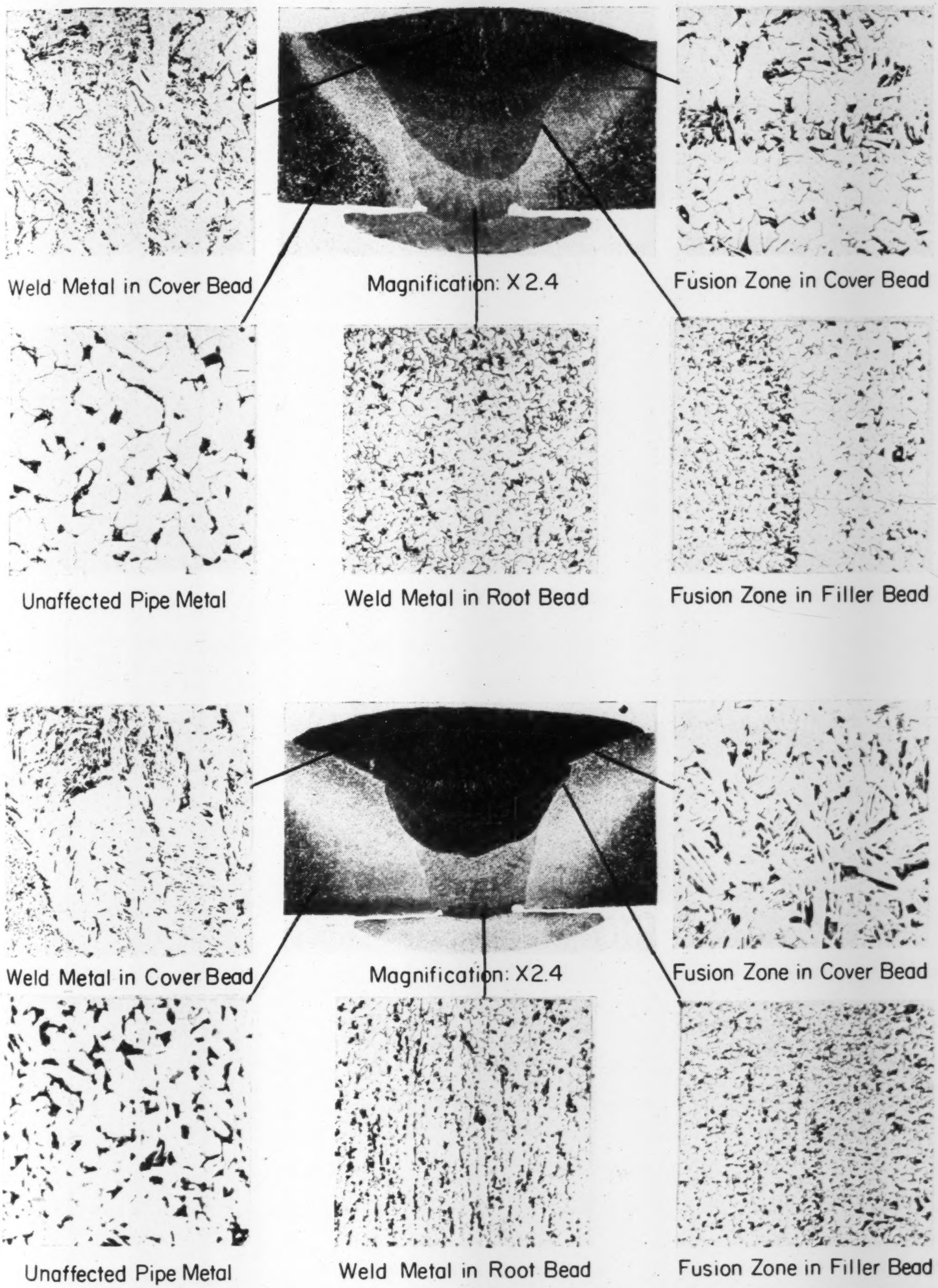
Weld Metal in Cover Bead
Magnification: X 2.4
Fusion Zone in Cover Bead
Unaffected Pipe Metal
Weld Metal in Root Bead
Fusion Zone in Filler Bead
Weld Metal in Cover Bead
Magnification: X2.4
Fusion Zone in Cover Bead
Unaffected Pipe Metal
Weld Metal in Root Bead
Fusion Zone in Filler Bead

FIG. 5.—Microstructure of Girth Welds—Capped Bessemer Steel (Above)—Grade A Killed Open-Hearth Steel (Below) (× 100). Reduced to about $\frac{7}{10}$ size in reproduction.

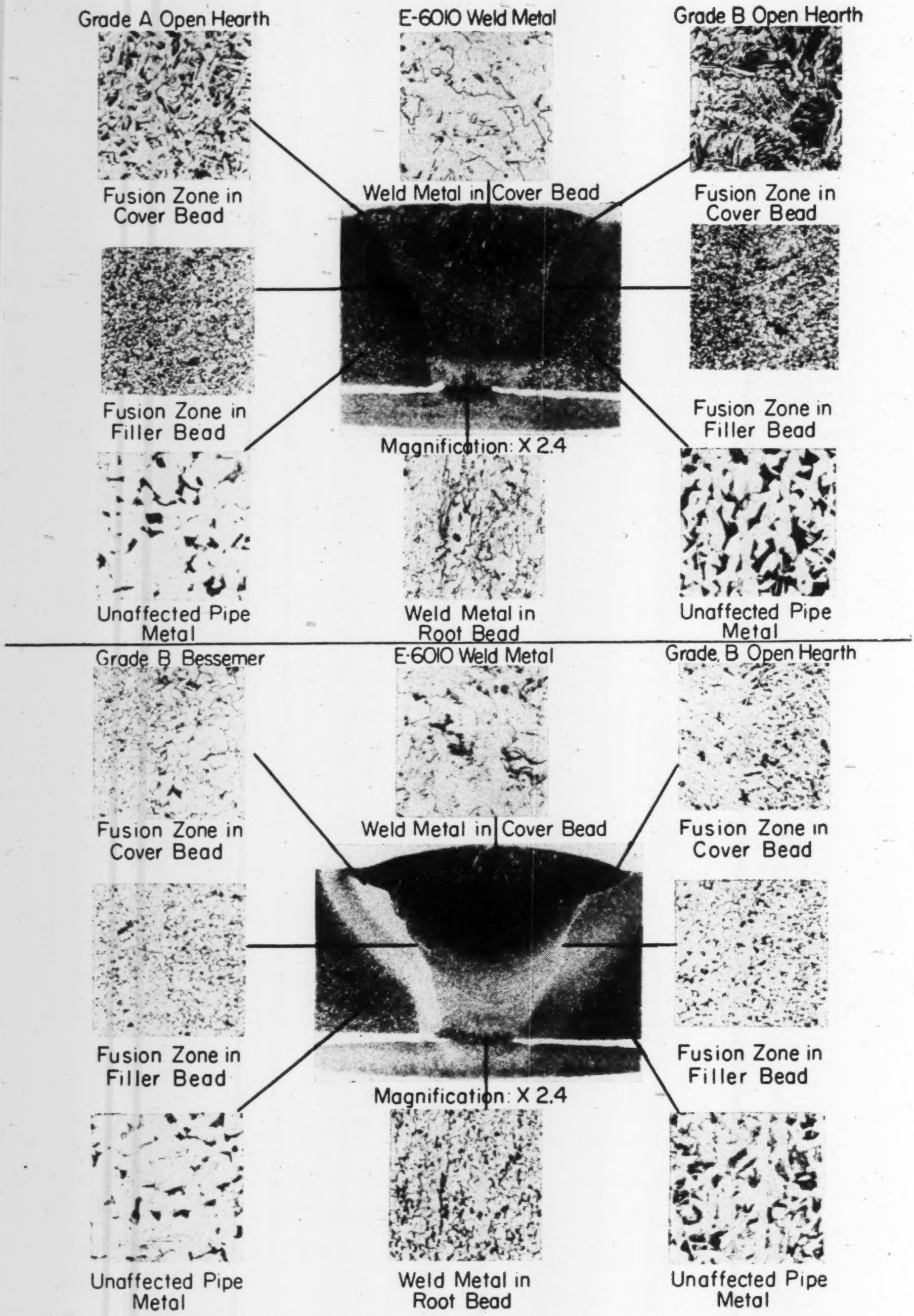
Grade A Open Hearth
E-6010 Weld Metal
Grade B Open Hearth
Fusion Zone in Cover Bead
Weld Metal in Cover Bead
Fusion Zone in Cover Bead
Fusion Zone in Filler Bead
Magnification: X 2.4
Fusion Zone in Filler Bead
Unaffected Pipe Metal
Weld Metal in Root Bead
Unaffected Pipe Metal
Grade B Bessemer
E-6010 Weld Metal
Grade B Open Hearth
Fusion Zone in Cover Bead
Weld Metal in Cover Bead
Fusion Zone in Cover Bead
Fusion Zone in Filler Bead
Magnification: X 2.4
Fusion Zone in Filler Bead
Unaffected Pipe Metal
Weld Metal in Root Bead
Unaffected Pipe Metal

FIG. 6.—Microstructure of Girth Welds—Grade A and Grade B Killed Open-Hearth Steels (Above)—Grade B Killed Bessemer and Grade B Killed Open-Hearth Steels (Below) (× 100). Reduced to about $\frac{7}{10}$ size in reproduction.

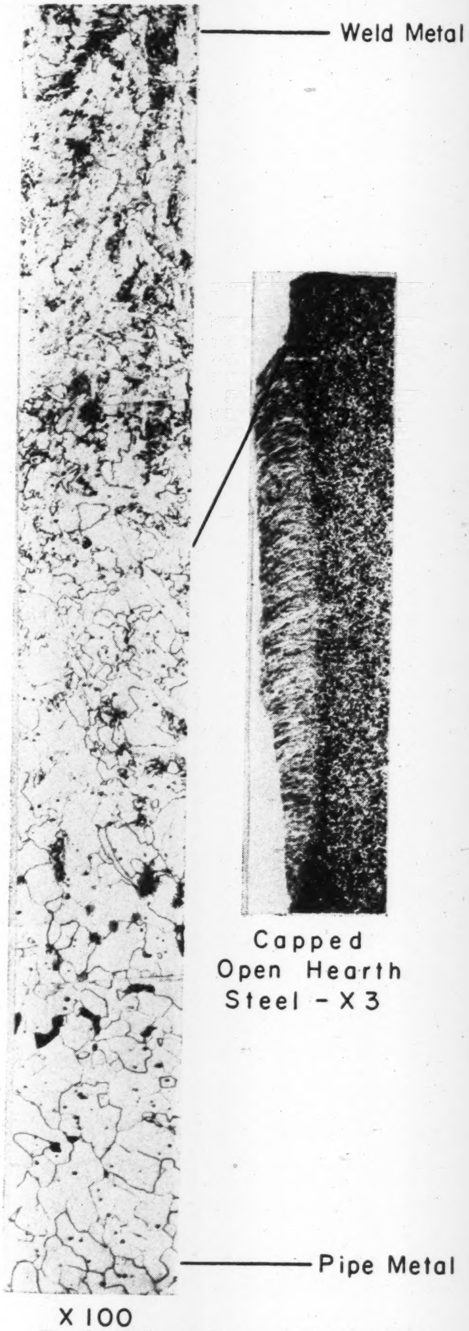

FIG. 7.—Microstructure of Cracking Susceptibility Specimens. Reduced to about $\frac{7}{16}$ size in reproduction.

2. After quench aging from 1250 F and 1700 F the killed steels were not embrittled. The capped steels after quenching from 1250 F had a lower level of toughness, but when quenched from 1700 F had a high level of toughness as determined by impact testing. The impact properties of all the steels after 2 per cent reduction by cold work and aging were lowered appreciably with the exception of capped bessemer steel which had a low impact strength before cold working.

3. The impact properties of capped steels compared with killed steels indicates the superior toughness characteristics of the killed steels. This was particularly observed in a comparison of the capped bessemer steel with the deoxidized bessemer seamless pipe.

4. The notch sensitivity in fatigue of the capped and killed steels was similar. The fatigue properties of the capped and killed bessemer steels and grade B killed open-hearth steels were similar with the exception of the aluminum-titanium killed open-hearth steel which had a lower fatigue strength. The capped open-hearth steels had a lower fatigue strength than the other steels.

5. All of the steel had comparable girth welding characteristics—girth welding of grade A to grade B seamless pipe or killed bessemer seamless pipe to killed open-hearth seamless pipe produced no unusual mechanical properties or metallographic structures. The grade B killed open-hearth steels were the only steels exhibiting cracks in the cracking susceptibility test. In the heat-affected zone of the bead weld hardness tests, the capped steels were lower in hardness compared with the killed steels.

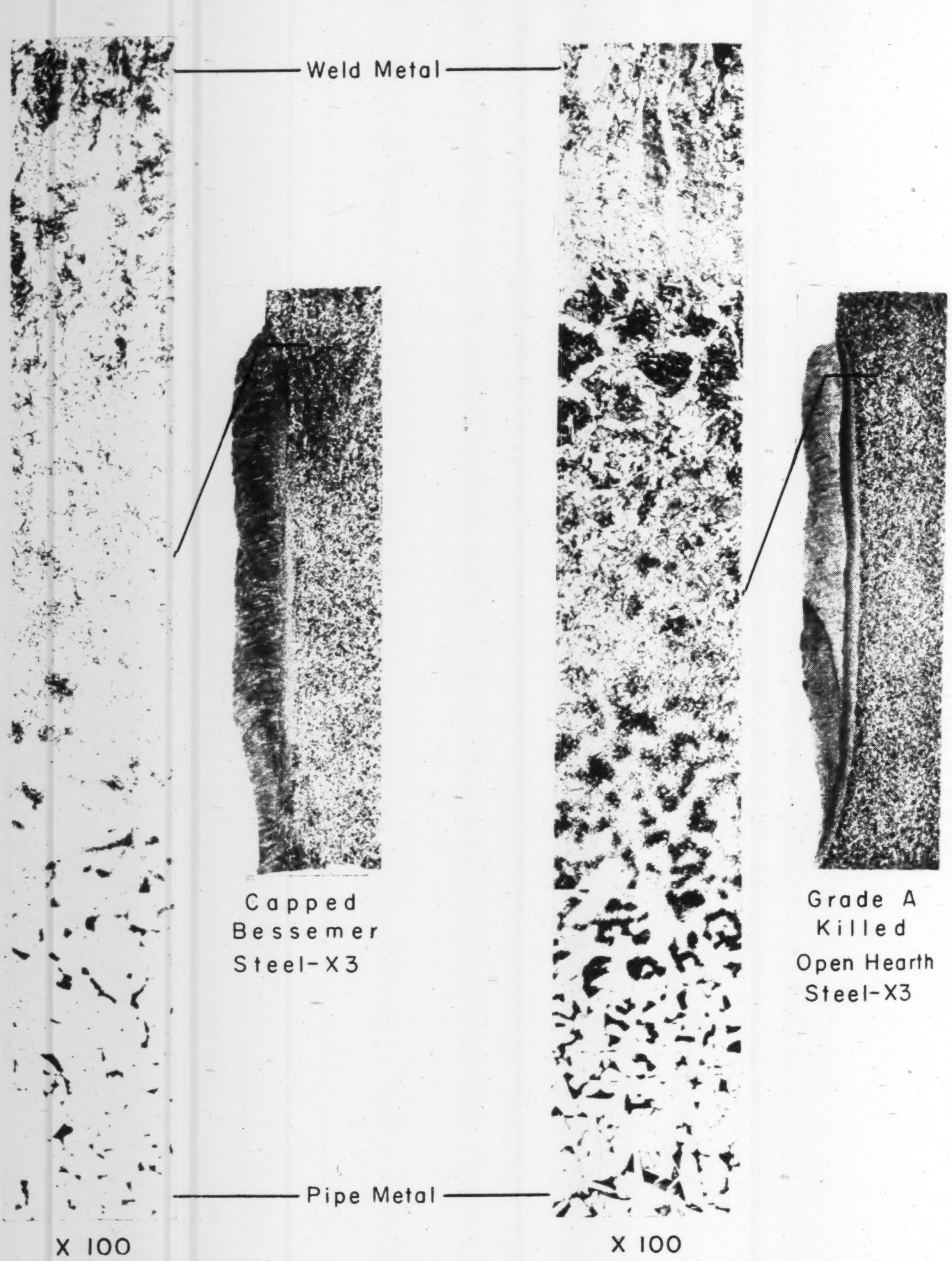
Weld Metal
Capped Bessemer Steel-X3
Grade A Killed Open Hearth Steel-X3
Pipe Metal
X 100
X 100

FIG. 8.—Microstructure of Cracking Susceptibility Specimens. Reduced to about $\frac{7}{10}$ size in reproduction.

## REFERENCES

(1) E. C. Wright, "Manufacture and Properties of Killed Bessemer Steel," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 158, p. 107 (1944).

(2) A. B. Wilder, "Physical Characteristics of Steel for Tubular Products," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 596 (1947).

(3) A. B. Wilder, W. B. Kennedy, and F. W Crouch, "Welding Characteristics of Open Hearth and Bessemer Seamless Steel Pipe," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 763 (1950).

(4) "The Making, Shaping and Treating of Steel," 6th Edition, United States Steel Corp. (1951).

(5) A. B. Wilder, "One Hundred Years of Bessemer Steelmaking," *Journal of Metals*, June, 1956, p. 742.

(6) A. H. Cottrell, "Dislocations and Plastic Flow in Crystals," Oxford University Press (1953).

(7) R. Rawlings and D. Tambini, "The Determination of the Alpha Phase Boundaries of the Iron-Nitrogen System by Internal Friction Methods," *Journal*, Iron and Steel Inst. (British), Vol. 184, p. 302 (1956).

## DISCUSSION

MR. NORMAN BLOCK.[1]—I am somewhat interested in the authors method for evaluating the notch toughness of the weld heat-affected zone. Could you elaborate somewhat on that?

MR. A. B. WILDER (*author*).—In testing the weld heat-affected zone, we are faced with the basic problem of trying to locate the notch in the heat-affected zone. This is a matter that can be discussed from various points of view. In our work we machined the test specimen so that the base of the notch would be in the heat-affected zone. The location of the notch is shown in a paper published in 1950. There would be some variation in these tests where we are trying to locate the notch in the heat-affected zone.

In any event, the impact values was very good in that zone.

MR. MAX B. HIGGINS.[2]—As I understand it, the tests were only conducted at temperatures up to 750 F. I was wondering what would the room temperature properties of these materials be after they had been in service for some 10,000 hr at 1000 to 1100 F.

MR. WILDER.—We have test information of material that has been subjected to aging tests at temperatures up to 1200 F and for 100,000 hr. This will be published in the future.

MR. HIGGINS.—That, of course, is something that is of prime importance because there has been used carbon steel operating lines in that temperature range and it would be interesting to know if there was any deterioration in the mechanical properties of the material at room temperature after having been in service in temperatures up to 1100 F for a reasonable length of time, I mean 10,000 to 20,000 hr.

MR. WILDER.—We have made tests of that nature, I do not have that information here.

MR. HIGGINS.—I would like to ask also about the heat-affected zone. Approximately what is the width of the heat-affected zone on this material after welding.

MR. WILDER.—I think it was about $\frac{1}{4}$ in. This would include the complete heat-affected zone, that is where you have the grain coarsening and also recrystallization. The photomicrographs in the paper show the extent of this zone.

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[1] Director of Materials Division, Foster Wheeler Corp., New York, N. Y.

[2] Consulting Engineer, Stamford, Conn.

MR. HIGGINS.—In other words, we could not expect the heat-affected zone to be more than $\frac{1}{4}$ in. wide for a carbon steel.

MR. WILDER.—I think it was about that.

MR. HIGGINS.—About the thicknesses involved.

MR. WILDER.—You can make that estimation from the photomicrographs, that is, knowing the magnification and the thickness of the material. This could be determined with a rule from the photomicrographs that are in the paper.

The question has been asked about the cracking susceptibility test. In that test we made a weld-bead under severe welding conditions and in the open-hearth material we did get cracking which was under the bead. This test has been described in a previous paper (Reference 3 of the paper). That cracking was rated at 28 per cent which in this test would indicate no difficulty in ordinary welding procedures. In other words, the cracking susceptibility test as described in the paper was so severe that you would have to get about 50 per cent cracking in order to be in danger, in this case the open-hearth steel had 28 per cent and all the other steels did not crack in the cracking susceptibility test.

# THE EFFECT OF VIBRATIONS ON THE STATIC YIELD STRENGTH OF A LOW-CARBON STEEL

By Gale E. Nevill, Jr.[1] and Franz R. Brotzen[1]

It has been known for some time that mechanical vibrations may affect certain physical properties. The early work by T. A. Read (**1, 2**)[2] indicates clearly that an interaction exists between certain lattice imperfections, such as dislocations, and applied periodic stresses. As a result of more recent internal-friction studies (**3**) three mechanisms by which dislocations may absorb energy from vibrations have been proposed:

(*a*) a resonance mechanism,

(*b*) a relaxation mechanism, and

(*c*) a mechanism of simple hysteresis.

Since dislocation movements are primarily responsible for plastic deformation of crystals, any mechanism by which dislocations could absorb energy and possibly move with greater ease would be of utmost interest.

The recently published work of Blaha and Langenecker (**4**) revealed a large (nearly 40 per cent) reduction in the steady stress required for yielding of zinc crystals due to low-amplitude vibration at 800 kc. This was believed indicative of an interaction between dislocations and the applied vibrations, such that the apparent stress necessary for plastic deformation was reduced. Blaha and Langenecker gave no explanation for their results, and no other work of a similar nature was known.

It was felt that an effort should be made to correlate any observed effect with known concepts of interaction between vibrations and lattice defects. Moreover, any phenomenon leading to a reduced static load-carrying capacity would deserve special attention.

A low-carbon steel was chosen for the present investigation because of its sharply defined yield point, importance as a structural material, and availability. The lower kilocycle range of frequencies was chosen because of the relative ease of obtaining vibrations of adequate intensity.

## EXPERIMENTAL APPARATUS

To stretch and vibrate the wire specimen,[3] a small tension-testing machine was constructed, Fig. 1. The lower head and vibrating mechanism, Fig. 2, was moved, through a series of reduction gears, at a constant rate of 0.05 in. per min by a $\frac{1}{4}$-hp motor. In the lower head was held, by means of adjustable setscrews, a $2\frac{3}{16}$ by $2\frac{3}{16}$ by $\frac{1}{2}$-in. barium-titanate crystal. Vibrations were transferred from the crystal to the specimen through a brass exponential stub. This stub was designed, according to the method given by Neppiras (**5**), to act as a device with amplitude-transformation ratio of approximately 10 to 1. The top of the stub and the specimen coupling were designed, as shown in Fig. 2, to insulate the wire electrically from the stub.

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[1] Dow Research Fellow in Mechanical Engineering and Associate Professor, respectively, Mechanical Engineering Department, The Rice Institute, Houston, Texas.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 758.

[3] 20-gage steel wire containing 0.05 per cent C, 0.41 per cent Mn, 0.004 per cent P, 0.023 per cent S, 0.02 per cent Si, annealed at 1400 F for $2\frac{1}{2}$ hr, furnace cooled.

The crystal-drive circuit, Fig. 3, supplied power to the crystal. The voltage across the crystal was measured with a vacuum-tube voltmeter.

The steady stress in the specimen was measured by a load arm with four Baldwin SR-4 strain gages wired in a bridge circuit, Fig. 4. Output from the bridge was connected directly to the recorder.

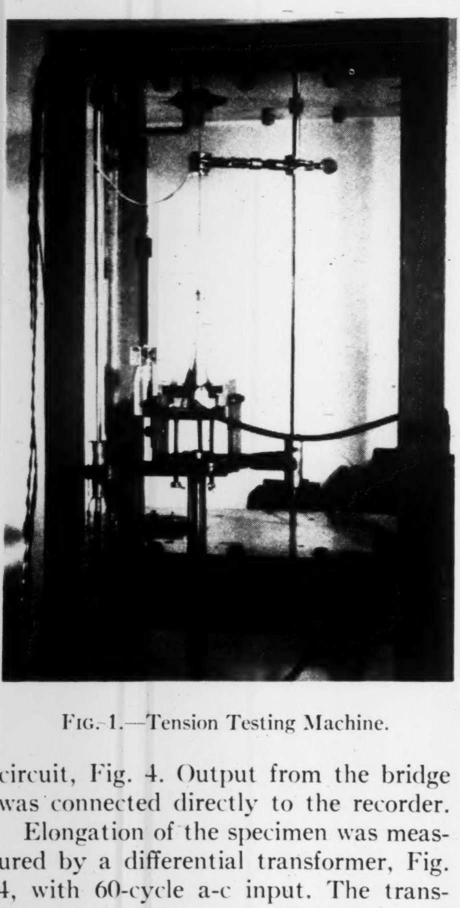

FIG. 1.—Tension Testing Machine.

Elongation of the specimen was measured by a differential transformer, Fig. 4, with 60-cycle a-c input. The transformer output was rectified, using a selenium rectifier, and connected to the recorder.

The vibrational amplitude was measured by taking advantage of the magnetostrictive effect. For the ferromagnetic specimen, assuming small periodic stresses, the change in magnetic flux through the wire was proportional to the elastic stress at any point (6). A small pickup coil was placed around the specimen wire and its leads connected to an oscilloscope. Since the voltage induced in the coil was proportional to the rate of change of flux, the amplitude of the oscilloscope pattern was proportional to the rate of change of stress. For sinusoidal vibrations, actually confirmed by the pattern on the oscilloscope screen, the stress amplitude was proportional to the amplitude on the oscilloscope divided by the frequency. Because the rate of change of flux was shown on the oscilloscope, only the desired alternating stress amplitude was observed and not the essentially steady load component. The amplitude of the oscilloscope pattern, divided by the

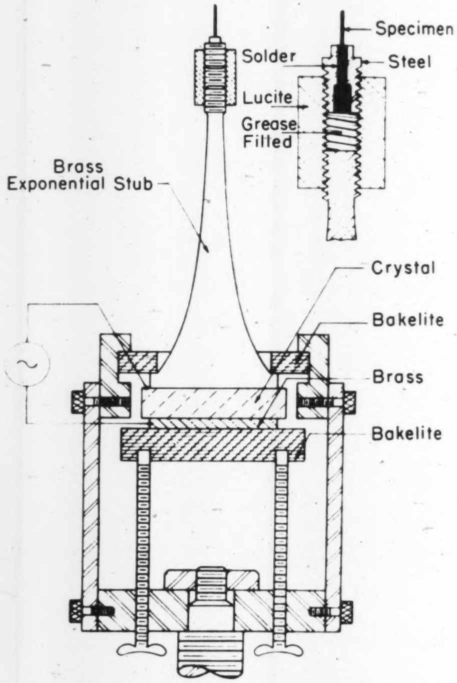

FIG. 2.—Detail of Lower Head Including Crystal Holder and Lower Specimen Coupling.

frequency, was therefore used to measure the vibrational stress amplitude in the specimen.

## EXPERIMENTAL PROCEDURE

The specimen wire was placed in the tension testing machine, passing through the pickup coil, Fig. 1. Under a small tension the wire was vibrated and the frequency adjusted until longitudinal standing waves were obtained in the specimen. The drive circuit was tuned to series resonance to obtain a large voltage across the crystal. The pickup coil was moved to the position of maximum vibrational amplitude on the wire. The specimen was then pulled and vibrated for short intervals at various amplitudes while the corresponding decreases in static yield stress were recorded. Stress-strain curves obtained were of the pattern shown in Fig. 5.

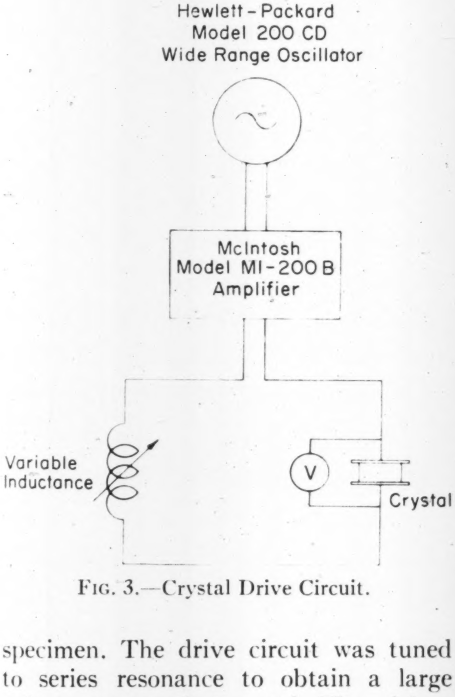
Hewlett-Packard
Model 200 CD
Wide Range Oscillator
McIntosh
Model MI-200B
Amplifier
Variable
Inductance
V
Crystal

FIG. 3.—Crystal Drive Circuit.

The effect of frequency was determined by adjusting the wire length so as to obtain standing waves over a range of frequencies. Amplitude-variation tests were then performed at various frequencies, as indicated above.

The influence of prior deformation on the strength reduction was examined by conducting amplitude-variation tests. These were repeated in each specimen at intervals until large values of plastic strain were attained.

The static yield-strength reduction was observed at various temperatures for a constant amplitude of vibration. The specimen temperature was varied by passing a direct current through the wire.

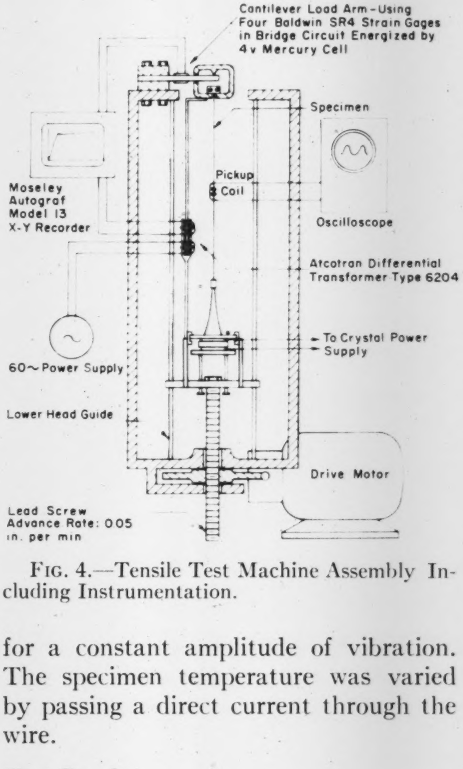
Cantilever Load Arm-Using
Four Baldwin SR4 Strain Gages
in Bridge Circuit Energized by
4 v Mercury Cell
Specimen
Moseley
Autograf
Model 13
X-Y Recorder
Pickup
Coil
Oscilloscope
Atcotran Differential
Transformer Type 6204
To Crystal Power
Supply
60~Power Supply
Lower Head Guide
Drive Motor
Lead Screw
Advance Rate: 005
in. per min

FIG. 4.—Tensile Test Machine Assembly Including Instrumentation.

### *Test Results:*

Results of the present investigation demonstrated that, when the specimen was subjected to vibration, the steady stress necessary to initiate plastic deformation was reduced, Fig. 5. This reduction proved to be proportional to the amplitude of vibration, Fig. 6. The drop in apparent yield strength was

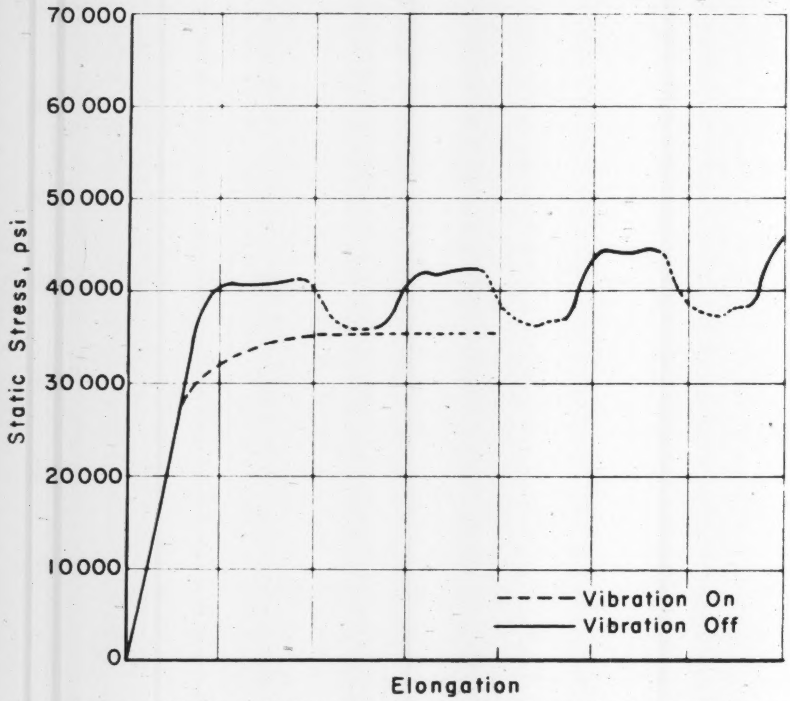

FIG. 5.—Effect of Mechanical Vibration on Static Yield Stress of a Low-Carbon Steel.

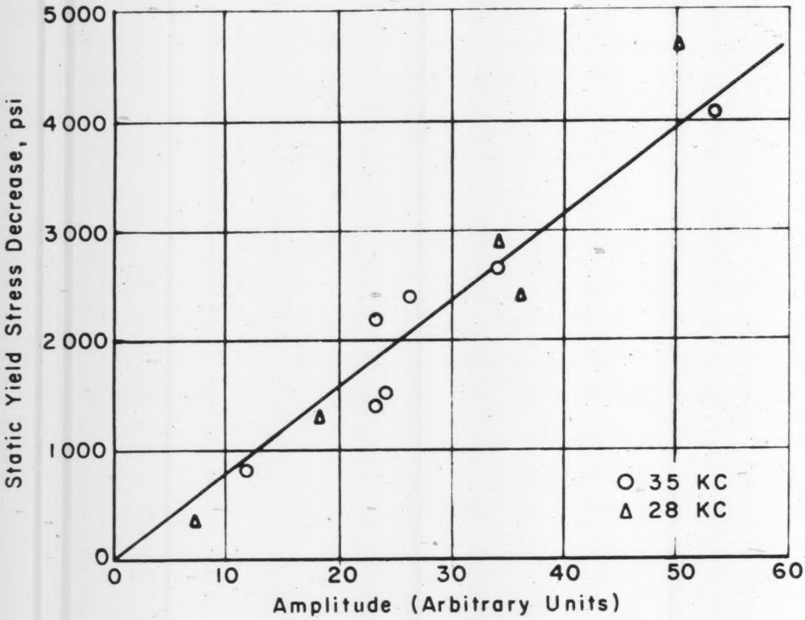

FIG. 6.—Effect of Amplitude of Vibration on Static Yield Stress Decrease at Room Temperature

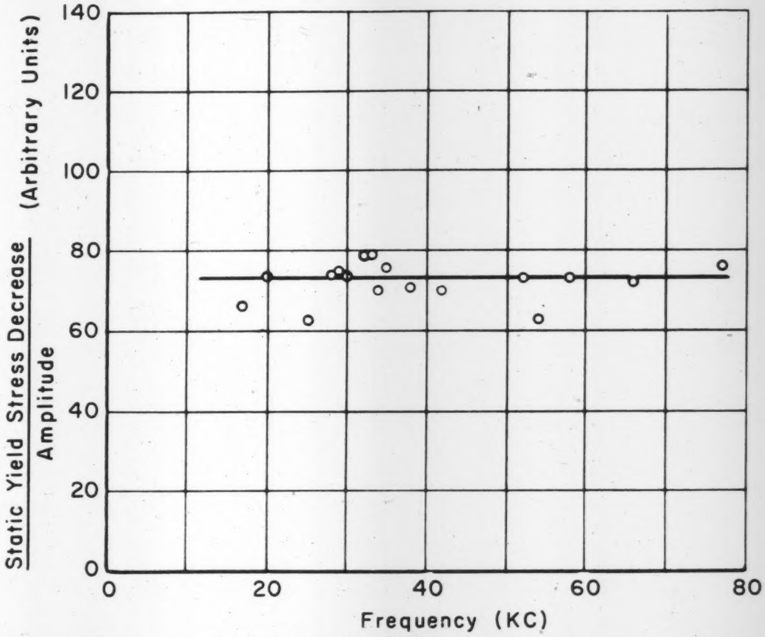

FIG. 7.—Effect of Frequency of Vibration on Static Yield Stress Decrease at Room Temperature.

found to be independent of frequency in the range of 15 kc to 80 kc, Fig. 7. Results proved also, Fig. 8, that the strength decrease was independent of prior strain for values of average permanent elongation up to 15 per cent. It was further demonstrated, Fig. 9, that in the temperature range between 30 and approximately 500 C the yield strength decrease was independent of temperature.

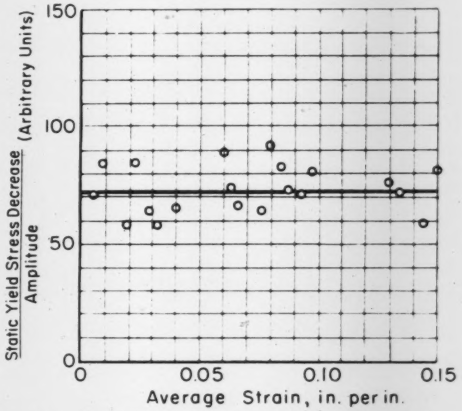

FIG. 8.—Effect of Permanent Strain on Static Yield Stress Decrease Due to Vibration at Room Temperature and 20 kc.

*Discussion:*

The concept of a yield strength in a crystal depends on the postulation of obstacles to either the movement or formation of dislocations. Since relatively small stresses are required for the formation of dislocations from a source, generation of dislocations is not the primary obstacle to large-scale plastic flow (**7,8**), except perhaps for high-purity single crystals. Since the specimens used in the present study were polycrystalline and contained numerous impurities, obstacles to the movement of dislocations may be assumed to be the primary source of strength. Any yield-strength decrease, therefore, would involve some mechanism by which dislocations can more easily break free from their obstacles.

One mechanism by which dislocations might absorb energy and overcome obstacles would be a resonance effect. It is known, however, that the natural frequency of a dislocation loop is in the order of $10^8$ sec$^{-1}$ (8). This frequency was not approached in this investigation, so that a resonance effect could not be the mechanism responsible for the observed phenomenon.

Another mechanism by which dislocations are able to absorb energy from an applied periodic stress is that of relaxation. If a dislocation oscillates at its natural frequency, $\nu_o$, it has a certain probability of surmounting its restraining energy hill of height $Q$. This probability, due to thermal activation, is $P = e^{-Q/KT}$, where $k$ is Boltzmann's constant and $T$ is the absolute temperature. The total number of successful jumps per unit time, or relaxation frequency, is $\nu_R = \nu_o e^{-Q/KT}$. The development of a relaxation model to explain the observed drop in yield strength due to applied vibration would require the existence of a characteristic relaxation-frequency spectrum. Since the experimental results showed no dependence on frequency or temperature, a relaxation effect must be discarded as a possible explanation.

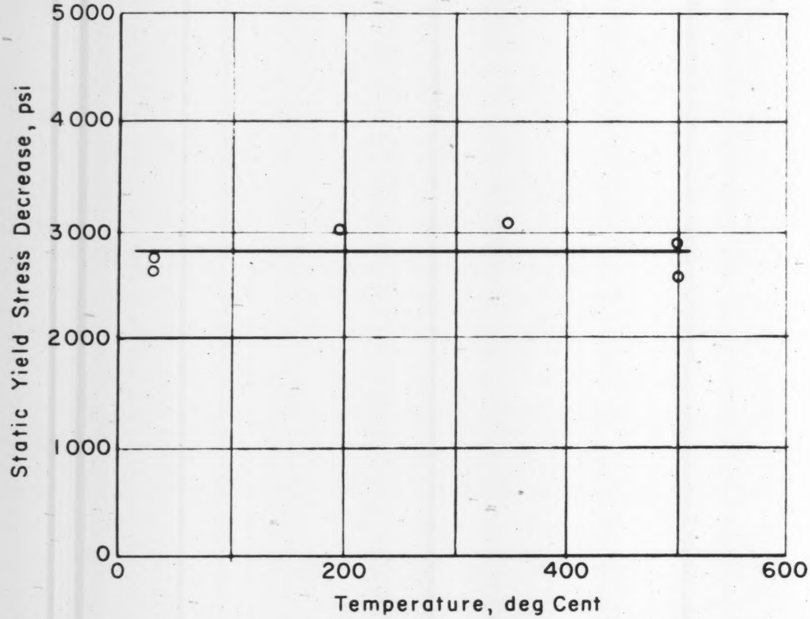

FIG. 9.—Effect of Temperature on Static Yield Stress Decrease Due to Vibration at Constan Amplitude and 20 kc.

Simple hysteresis is another mechanism by which dislocations absorb energy from applied periodic stresses (9). This energy-absorption mechanism, however, is not suited for an interpretation of a decrease in yield strength.

An explanation of the observed results can be made by considering a macroscopic superposition of steady and alternating stresses. Consider the specimen under a steady uniaxial stress, $S$, on which is superposed a plane longitudinal elastic wave of maximum stress amplitude, $A$. Consider also that a minimum stress, $S_y$, is necessary to produce yielding. For the case of $S_y > S + A$, the specimen will not yield. For the case of $S_y < S + A$, however, the specimen for a portion of the vibrational cycle is subjected to a total stress greater than that required to induce plastic deformation. This superposition of stresses is schematically illustrated in Fig. 10.

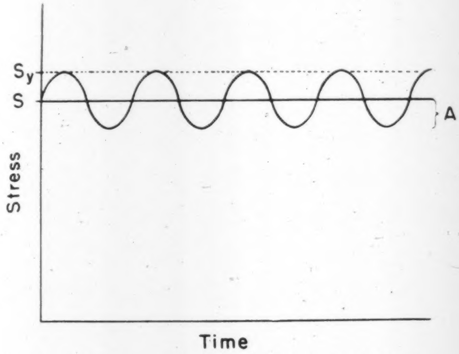

FIG. 10.—Superposition of Steady and Alternating Stresses to Cause Yielding.

From a sub-microscopic point of view, a dislocation is raised part-way up its restraining stress hill by the steady stress. When the alternating stress becomes sufficiently high, the dislocation overcomes its initial restraining hill and possibly moves further across a number of obstacles, causing plastic deformation. The dislocation does not return to its initial position when the alternating stress is reversed but only moves to a lower stress position in front of its new obstacle. When, due to the periodic stress, the dislocation is again activated, it travels in its initial direction producing more plastic deformation. This procedure is repeated with each stress cycle, provided $S + A > S_y$. Earlier in this paper mention was made of results obtained by Blaha and Langenecker (**4**) with zinc single crystals. It is conceivable that their findings could also be interpreted in terms of a stress superposition.

The mechanism of stress superposition does not conflict with the delayed yield phenomenon observed in low-carbon steels. The delayed yield was shown by Vreeland *et al* (**10**) to be a function of the cumulative time at stress, so that the delayed yield effect might postpone but not prevent the beginning of vibration-induced plastic deformation.

It is interesting that under continuous vibration the specimen does not seem to maintain as sharp a yield point as observed in the absence of vibrations, Fig. 5. At stresses below the yield point, that is, in the truly elastic range, vibrations do not change the stress-strain relation.

The picture of a superposition of steady and alternating stresses is compatible with the observed linear-amplitude dependence and frequency independence of the yield-strength decrease. It is further seen that this superposition agrees with the observed independence of temperature and prior strain. Although $S_y$ would be changed by strain hardening or temperature variation, the difference, $A$, between $S_y$ and $S$ would remain constant.

It should be mentioned that, since standing waves occurred in the specimen, the strain was not homogeneous. This means that the independence of prior deformation shown by the static yield-strength decrease actually extends to larger values of strain than those indicated in Fig. 8.

## SUMMARY

In the frequency range of 15 kc to 80 kc, applied mechanical vibrations reduce the steady stress necessary to initiate yielding in tension in a low-carbon steel specimen. This decrease in yield strength is proportional to the vibrational amplitude. It is independent, however, of the frequency of vibration, temperature, and prior strain. This phenomenon is attributed to a mechanism of superposition of steady and alternating stresses.

*Acknowledgment:*

The authors wish to acknowledge the cooperation of the Office of Scientific Research, United States Air Force, Contract AF-49-(638)-78, under whose sponsorship this research was carried out.

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## REFERENCES

(1) T. A. Read, "Internal Friction of Single Metal Crystals," *Physical Review*, Vol. 58, p. 371 (1940).

(2) T. A. Read, "Internal Friction of Single Crystals of Copper and Zinc," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 143, p. 30 (1941).

(3) W. Kempe and E. Kroner, "Dislocation Damping of Aluminum Single Crystals at Room Temperature," *Zeitschrift für Metallkunde*, Vol. 47, No. 5, p. 302 (1956).

(4) F. Blaha and B. Langenecker, "Tensile Deformation of Zinc Crystals Under Ultrasonic Vibration," *Naturwissenschaften*, Vol. 42, p. 556 (1955).

(5) E. A. Neppiras, "A High-Frequency Reciprocating Drill," *Journal of Scientific Instruments*, Vol. 30, p. 72 (1953).

(6) R. M. Bozorth, "Ferromagnetism," D. Van Nostrand Co., Inc., New York, N. Y. (1951).

(7) N. F. Mott and F. R. N. Nabarro, "Dislocation Theory and Transient Creep," *Report of a Conference on Strength of Solids*, Physical Society, London (1948).

(8) A. H. Cottrell, "Dislocations and Plastic Flow in Crystals," Oxford at the Clarendon Press, London (1953).

(9) A. S. Nowick, "Internal Friction in Metals," *Progress in Metal Physics*, Vol. 4, Pergamon Press, Ltd., London (1953).

(10) T. Vreeland, Jr., D. S. Wood, and D. S. Clark, "A Study of the Mechanism of the Delayed Yield Phenomenon," *Transactions*, Am. Soc. Metals, Vol. 45, p. 620 (1953).

# ELECTRICAL RESISTIVITY AND THERMOELECTRIC POTENTIAL OF RHENIUM METAL*

By Gordon B. Gaines and Chester T. Sims[1]

## Synopsis

The electrical resistivity of rhenium with an impurity content of approximately 0.06 per cent can be represented with an error of less than 1 per cent of the measured values between room temperature and 2600 deg Kelvin by the equation:

$$\rho = -7.25 + 9.88 \times 10^{-2}\,T - 3.10 \times 10^{-5}T^2 + 4.0 \times 10^{-9}T^3,$$

where: $\rho$ is in microhm-cm and $T$ is in deg Kelvin. The 20 C value is 19.3 microhm-cm. The temperature coefficient of resistivity, $\alpha_{20C}$, decreases from 4.02 per deg C at 100 C to 2.06 per deg C at 2300 C. At 25 C, the resistivities of platinum-1 per cent rhenium and platinum-2 per cent rhenium alloys were found to be $19.9 \times 10^{-6}$ and $25.8 \times 10^{-6}$ ohm-cm, respectively.

The thermoelectric potentials generated by rhenium-molybdenum, rhenium-tungsten, and rhenium-tantalum junctions were measured over the temperature range from 870 to 2490 C. Rhenium-tungsten and rhenium-molybdenum junctions show promise as thermocouples up to 2600 C in neutral or reducing atmosphere. The behavior of the rhenium-tantalum junction is peculiar and apparently is of no practical use as a thermocouple. Between 870 and 1720 C, the thermoelectric force of the rhenium-tungsten junction can be represented by the equation:

$$E = 0.015T - 5.4.$$

Between 1720 and 2400 C, the approximate equation is:

$$E = 0.0093T + 4.4.$$

For the rhenium-molybdenum junction between 870 and 1500 C, the data are represented by the equation:

$$E = 23.9 - \frac{1250}{T}.$$

From 1500 to 2600 C, the approximate equation is:

$$E = 0.0055T + 7.3.$$

In the above equations, $E$ is in mv and $T$ is in deg Cent. Platinum-platinum 1.0 rhenium and platinum-platinum 2.0 rhenium junctions were investigated up to 1300 C and the results were found to agree generally with earlier work.

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[1] Assistant Chief, Solid State Devices Division and Assistant Chief, Nonferrous Physical Metallurgy Division, respectively, Battelle Memorial Institute, Columbus, Ohio.

Rhenium metal is of interest for use as a construction material in electrical and electronic applications. Recent investigations of the properties of rhenium have shown that it melts at 3180 C, has excellent room- and high-temperature strength properties, and can be fabricated into thin sheet and wire (1).[2] In addition, rhenium forms a conducting oxide, $ReO_3$, has excellent resistance to the water cycle, and does not form a carbide (2, 3). As a portion of the current investigations of rhenium, its electrical resistivity and thermoelectric properties have been studied and are reported in this paper.

The first measurement of the electrical resistivity of rhenium was made in 1930 by Agte and co-workers (4), who reported a value of $21.1 \times 10^{-6}$ ohm - cm ($\pm 15$ per cent) at 20 C. The temperature coefficient of resistivity from 0 to 100 C was given as $3.11 \times 10^{-3}$ per deg Cent. Meissner and Voigt (5) found a value of $18.9 \times 10^{-6}$ ohm - cm for the electrical resistivity at 0.16 C. Extended to 20 C by use of Agte's temperature coefficient, the latter value becomes $19.0 \times 10^{-6}$ ohm - cm. Sims *et al* reported (1) a value of $19.4 \pm 0.25 \times 10^{-6}$ ohm - cm in 1954. This value was an average of numerous determinations on drawn and annealed wire, ranging in diameter from about 0.03 to 0.06 in. Chemical analysis of rhenium metal prepared by the methods then known showed that it contained 0.2 per cent total impurities. This relatively high impurity content was a result of the first fabrication processes developed in the current work. In addition, subsequent resistivity determinations on the same and similar wire stock failed to reproduce the initial results. Following the development of improved consolidation and fabrication practices, which resulted in rhenium wire of higher purity, a new series of resistivity measurements was completed at room temperature and then extended to elevated temperatures. These results are reported in detail in this study. Resistivity values for rhenium - platinum alloys are also included.

In recent work (6), it was shown that rhenium metal produces a sizable and positive emf against platinum which can be represented by the equation:

$$E = 1.56 - 9.0 \times 10^{-3}\, T + 1.29 \times 10^{-5}\, T^2$$

where $E$ is the potential difference in mv and $T$ is the temperature in deg Kelvin. The equation:

$$P = 9.0 \times 10^{-3} + 2.58 \times 10^{-5}\, T,$$

gives the thermoelectric power, $P$, in mv per deg. Rhenium produces a potential difference of approximately 29 mv against platinum at 1600 C. Since tungsten *versus* platinum and molybdenum *versus* platinum both produce approximately 55 mv at this temperature, rhenium - tungsten or rhenium - molybdenum thermocouples would be expected to produce about 25 mv at the same temperature. Such thermocouples would be interesting because of their potential use at very high temperatures. Accordingly, the thermoelectric potentials as a function of temperature were established for thermocouples of rhenium *versus* molybdenum *versus* tungsten, and *versus* tantalum.

Schulze (7) reported the thermoelectric potential generated by platinum-rhenium alloys *versus* platinum. He found that at 1200 C a platinum - 5 per cent rhenium alloy *versus* platinum generated about 28 mv. A platinum - 10 per cent rhenium alloy *versus* platinum generated about 33 mv at the same temperature. In the present work, Schulze's measurements were repeated for arc-melted platinum-rhenium alloys containing up to 2 per cent rhenium fabricated into wire.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 768.

TABLE I.—SPECTROCHEMICAL ANALYSIS OF RHENIUM RESISTIVITY SPECIMEN.[a]

| Impurity | Per Cent by Weight |
|---|---|
| Copper | 0.0005 |
| Tin | 0.014 |
| Aluminum | 0.015 |
| Silicon | 0.010 |
| Magnesium | 0.005 |
| Calcium | 0.010 |
| Molybdenum | 0.004 |
| Gold | 0.0$x$ |
| Total | 0.0585<br>(plus gold) |

Elements checked but not found: Arsenic, antimony, barium, beryllium, bismuth, boron, cadmium, chromium, cobalt, gallium, germanium, lead, manganese, niobium, nickel, platinum, potassium, silver, sodium, strontium, tellurium, titanium, tungsten, vanadium, zinc, zirconium.

[a] Analysis by W. B. Coleman and Co., Philadelphia, Pa.

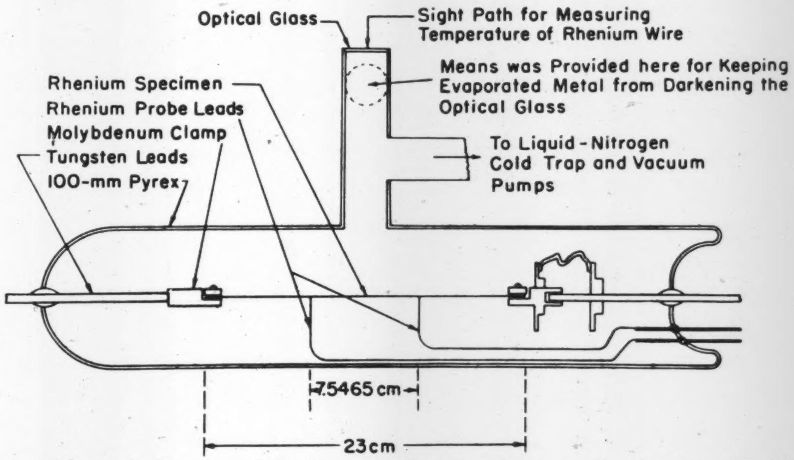
Optical Glass
Sight Path for Measuring Temperature of Rhenium Wire
Means was Provided here for Keeping Evaporated Metal from Darkening the Optical Glass
Rhenium Specimen
Rhenium Probe Leads
Molybdenum Clamp
Tungsten Leads
100-mm Pyrex
To Liquid-Nitrogen Cold Trap and Vacuum Pumps
7.5465 cm
23cm

FIG. 1.—Schematic Drawing of the Vacuum Chamber Employed for the Rhenium Resistivity Measurements.

## ELECTRICAL RESISTIVITY STUDIES

The electrical resistivity of a rhenium wire was determined over the temperature range from room to 2600 K. The wire was prepared as follows. Ammonium perrhenate, which had been ground by hand in an agate mortar to insure minimum impurity pickup was hydrogen reduced to rhenium-metal powder. This was consolidated by resistance sintering. The sintered bar was then reduced to a wire 23 cm long by swaging and wire drawing and annealed at 1750 C. Spectrochemical analysis of a section of this wire, made after the resistivity measurements were taken, showed that the impurity content was approximately 0.06 per cent by weight (see Table I).

The resistivity was calculated from the measured resistance by the well-known relationship:

$$\rho = \frac{RA}{l} \quad \ldots\ldots\ldots\ldots\ldots (1)$$

where $\rho$ is the resistivity in ohm-cm, $R$ is the measured resistance in ohms, $A$ is the cross-sectional area of the wire in cm$^2$ and $l$ is the length in cm. The wire diameter, averaged from 50 individual micrometer measurements, was 0.0518 in. The measured resistances were those of a 7-cm center section of the 23-cm wire.

## RESISTIVITY MEASUREMENT METHODS

The resistivity was determined as a function of temperature in several distinct temperature ranges using different experimental techniques. The method used for each temperature range is given in the order of increasing temperature, although chronologically the two highest temperature ranges were studied immediately after the room-temperature measurements. Results of the measurements are given following discussion of the experimental methods.

### *Room Temperature:*

The room-temperature resistivity was determined first. Resistance of the measured length of wire was determined in a constant-temperature room at 77 F with a Kelvin bridge. In order to assure that the resistivity obtained was for wire in the annealed condition, the resistance was determined after various annealing times up to seven hours. After three hours at the annealing temperature of 1750 C, resistance values were constant within the experimental error. From earlier work, it was found that 25 per cent cold work raised the resistivity by about $2 \times 10^{-6}$ ohm-cm (2).

### *Room Temperature to 725 K:*

The arrangement shown in Fig. 1 was used in this temperature range, although the sight tube shown was replaced by a seal through which thermocouple leads were passed. The entire apparatus was heated in a large muffle furnace. Chromel-alumel thermocouples were used to determine the temperature. Resistance was measured with a portable Leeds & Northrup Kelvin bridge.

### *630 to 1175 K:*

The pyrex vacuum-sealed apparatus of Fig. 1 was not used in this temperature range. Instead, the rhenium wire was mounted in an open alumina boat and heated in a hydrogen-atmosphere molybdenum-wound tube furnace. Chromel-alumel thermocouples were used in determining the temperature. Due to a small temperature gradient along the wire, slightly different resistance values were found when the probe leads were reversed at the Kelvin bridge. Thus, for each temperature, the leads were reversed and the average of the two resistance readings was used. The resistivity values calculated from this procedure fall on a smooth curve connecting the lower and higher temperature values determined by other methods. Apparently the temperature gradient is not serious within the accuracy of the plot of the data, shown later.

### *1100 to 2050 K:*

In this range, the wire was heated by its self resistance; the resistivity was calculated from the wire dimensions and the voltage across and the direct current through the measured length. The voltage was measured with a type K2 potentiometer. The current was determined by measuring the voltage across a standard resistor (NBS, calibrated) in series with the rhenium wire. A second type K2 potentiometer was used in the latter voltage measurements. The two potentiometers were balanced simultaneously and standardized from the same standard cell. The temperature was measured with a calibrated Leeds & Northrup optical pyrometer; an average was taken of several readings by two observers. A temperature gradient within the sensitivity of the optical pyrometer was not detected. The spectral emissivity ($\lambda = 0.65\ \mu$) was taken from earlier work in this investigation as 0.42 (2) over the temperature range employed.

*2000 to 2600 K:*

In this range, the resistance of the measured length of the rhenium specimen was determined, as above, from the voltage across and the direct current through the measured length. A battery power source was not available that would supply sufficient current for temperatures above 2000 K, so a direct current source consisting of a generator-battery combination was employed. The short-time voltage fluctuations of this source were greater than those in the simple battery supply used for the measurements in the 1100 to 2050 K range. The voltage across the specimen was determined with a $\frac{1}{2}$ per cent meter; the current and temperature were determined as above.

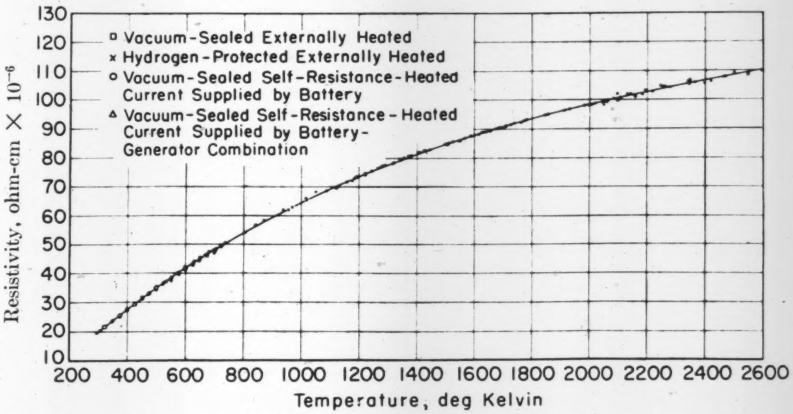

FIG. 2.—The Electrical Resistivity of Rhenium as a Function of Temperature.

At these high temperatures, a deposition of metal on the glass walls by evaporation from the filaments became evident. The data were corrected for the resulting change in diameter by employing the known resistivity values for the temperature range, 1100 to 2050 K, where evaporation is not significant. The procedure for determining this correction was as follows: the rhenium wire was heated to a selected temperature in the 2000 to 2600 K range, and the voltage across the measured length and current determined with as little loss of time as possible. Immediately after these measurements, the temperature was dropped to below 2000 K. Several measurements were then taken from 1100 to 2050 K, and a smooth curve drawn through the points. From this curve and the relationship between resistivity and temperature in this range as determined previously, the change in diameter was calculated. This procedure was repeated until a number of corrected resistivity determinations had been made from 2000 to 2600 K. The actual total correction for change in diameter was very small (<1 per cent).

*Resistivity Results:*

All the electrical resistivity data are plotted in Fig. 2. The temperature ranges covered by the various methods discussed above are shown by different designation of points on the graph. The value for 20 C, requiring a small extrapolation from this work, is $19.3 \times 10^{-6}$ ohm-cm. For convenience, the resistivities for every 200 C are given in Table II. These values were taken from the "best" smooth curve (not shown) drawn through the data shown in Fig. 2. The drawn curve in Fig. 2 is the equation:

$$\rho = -7.25 + 9.88 \times 10^{-2}T - 3.10 \times 10^{-5}T^2 + 4.0 \times 10^{-9}T^3 \ldots (2)$$

$T$ is the temperature in degrees Kelvin, and $\rho$ is the resistivity in microhm-cm.

TABLE II.—TEMPERATURE COEFFICIENTS OF RESISTIVITY CALCULATED FROM THE PRESENT WORK AND COMPARED TO THOSE OF AGTE *et al* (**4**).

| Temperature, $T$ | | Resistivity, $\rho$ microhm-cm | | Resistivity-Temperature Coefficient per deg Cent, $\alpha = \dfrac{\frac{R_T}{R_{293}} - 1}{T^a - 293} \times 10^3$ | |
|---|---|---|---|---|---|
| deg Cent | deg Kelvin | Present Work | Agte *et al* | Present Work | Agte *et al* |
| −190 | 83 | | 4.94 | | 3.65 |
| −30 | 243 | | 15.4 | | 3.35 |
| 0 | 273 | | 19.8 | | 3.11 |
| 20 | 293 | 19.3 | 21.1 | | 3.11 |
| 100 | 373 | 25.5 | | 4.02 | |
| 120 | 393 | | 26.1 | | 3.11 |
| 300 | 573 | 39.8 | | 3.79 | |
| 500 | 773 | 52.3 | | 3.56 | |
| 700 | 973 | 63.3 | | 3.35 | |
| 900 | 1173 | 72.8 | | 3.15 | |
| 1100 | 1373 | 80.6 | | 2.94 | |
| 1300 | 1573 | 87.3 | | 2.75 | |
| 1500 | 1773 | 92.8 | | 2.57 | |
| 1700 | 1973 | 97.8 | | 2.42 | |
| 1900 | 2173 | 102.1 | | 2.28 | |
| 2100 | 2373 | 106.1 | | 2.16 | |
| 2222 | 2495 | | 125 | | 2.23 |
| 2300 | 2573 | 110.0 | | 2.06 | |
| 2417 | 2690 | | 130 | | 2.14 |
| 2712 | 2985 | | 134 | | 1.98 |

[a] In degrees Kelvin.

The maximum difference between the best curve and the one represented by Eq 2 is less than 1 per cent.

Agte *et al* (4) have reported the temperature coefficient of resistivity, $\alpha$ (20 C), for several temperatures in the ranges from −190 to 120 C and 2222 to 2712 C. From the present work, the temperature coefficient of resistivity has been calculated for temperatures from 20 to 2300 C at 200 C intervals. These data are compared to Agte's results in Table II, where it can be seen that the present work gives higher values at the lower temperatures and lower values at the very high temperatures.

## PLATINUM-RHENIUM ALLOY RESISTIVITY MEASUREMENTS

Alloys of platinum containing 1, 2, and 5 per cent rhenium were prepared by arc melting on a water-cooled copper hearth under argon. The 1 and 2 per cent rhenium alloys were found to be fabricable to 0.020-in. wire. Following determination of the thermoelectric potential of these alloys against pure platinum as described later, electrical resistivity was determined at room temperature.

Determination of the electrical resistivity of 27.75-cm lengths of the alloy wires together with a 0.020-in. platinum wire (thermocouple grade) included as a standard was made on a laboratory-type Leeds & Northrup Kelvin bridge. The resistance measurements were conducted at a constant temperature of 25 C, and resistivity calculated by Eq 1. Results are given in Table III. The value found for pure platinum at 25 C was 11.0 × $10^{-6}$ ohm-cm. The literature value for platinum at that temperature is 10.94 microhm-cm, giving an indication of the accuracy of the resistivity values for the platinum-rhenium alloys. The sizable increase in electrical resistivity caused by rhenium additions to platinum are of interest where platinum alloys of high resistivity are desirable.

TABLE III.—ELECTRICAL RESISTIVITY OF RHENIUM-PLATINUM ALLOYS AT 25 C.

| | Resistivity, microhm-cm |
|---|---|
| Platinum (present work) | 11.0 |
| Platinum (literature) (**8**) | 10.94 |
| Platinum - 1.0 rhenium | 19.9 |
| Platinum - 2.0 rhenium | 25.8 |

## THERMOELECTRIC POTENTIAL STUDIES

Exploratory studies of the thermoelectric potentials generated by rhenium-molybdenum, rhenium-tungsten, and rhenium-tantalum junctions were conducted for the temperature range 870–2490 C. In addition, the thermoelectric potentials produced by platinum-1 and 2 per cent rhenium alloys *versus* platinum were measured for comparison with earlier data.

### RHENIUM *versus* MOLYBDENUM, TUNGSTEN, AND TANTALUM

Individual thermocouples were prepared for each combination measured. The rhenium wire, 0.020-in. in diameter, was drawn by procedures described previously (1). All measurements were conducted simultaneously as described below. The results are discussed following description of the measurement methods.

*Thermoelectric Potential Measurement Method:*

Individual thermocouples, 20-in. long, were made using rhenium wire welded to 0.015- or 0.020-in. diameter molybdenum, tungsten, and tantalum wire. The welds were made in a helium atmosphere to insure properly fused beads at the junctions.

The temperature *versus* electrical potential relationships produced by the three couples were measured in a resistance sintering bell, normally used to vacuum sinter refractory metals. The pressure varied from 0.4 to 4 mm of mercury. The three thermocouple beads were inserted into small holes drilled in the surface of a $\frac{3}{4}$-in. molybdenum rod, 8 in. long, mounted vertically in the sintering apparatus. The three holes were grouped on a circumference of the rod near the center of its length. A fourth hole, located nearby was used as a blackbody light source for optical determination of the true temperature of the rod. The blackbody hole was 8 times as deep as its diameter, and was viewed through a sight hole in the sintering bell. The thermocouple leads, separated to avoid electrical shorts, passed through an insulating seal in the sintering bell floor to a cold junction. Potential measurements were taken on a semi-precision Leeds & Northrup potentiometer.

The temperature of the thermocouple beads was increased by self-resistance heating the molybdenum rod. Thermoelectric potentials were determined for temperatures from 780 C up to 2500 C, the useful temperature limit of the molybdenum rod. Temperature corrections for spectral absorption by the sintering-bell sight glass amounted to 20 C at 1500 C and 30 C at 2300 C. A linear correction based on these values was applied to the temperatures recorded.

*Thermoelectric Potential Measurement Results:*

The emf values plotted against temperature for molybdenum, tantalum, and tungsten against rhenium are shown in Fig. 3. The thermoelectric force and thermoelectric powers of the rhenium-tungsten and the rhenium-molybdenum couples are rather large. The temperature-emf relationship of the rhenium-tantalum couple is peculiar, may be unstable with time at a given temperature, and apparently has no practical value.

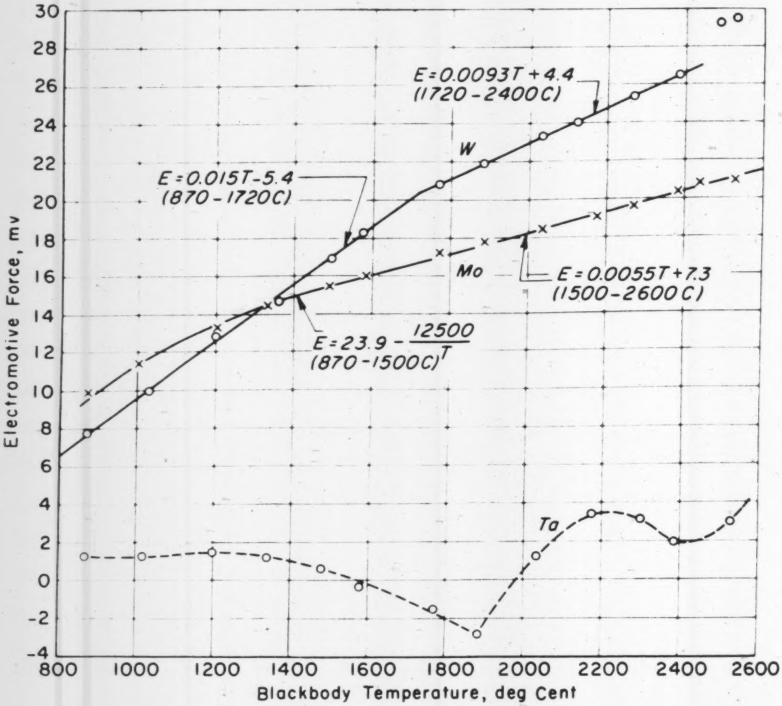

FIG. 3.—The emf for Rhenium Couples with Tungsten, Molybdenum, and Tantalum.

The experimental data for the rhenium-tungsten couple can be represented most simply by two straight lines that meet at about 20.5 mv and 1720 C. The rhenium-molybdenum system is represented by a curved line at low temperatures; at higher temperatures, it is represented by a straight line tangent to the curve at about 15.5 mv and 1500 C. The mathematical expressions relating the thermal emf as a function of temperature, and the temperature ranges for which these expressions apply, are also shown in Fig. 3. The thermoelectric data are summarized in Table IV.

The rhenium-tungsten thermocouple is potentially useful at temperatures up to about 2800 C. A rhenium-tungsten eutectic is thought to occur at about 2825 C (**9**), precluding use above this temperature. No similar information is available on the rhenium-molybdenum system, except that no melting appeared to have occurred in the couple bead when the junction was operated at 2500 C. However, the thermal emf of both couples varied from the straight-line relationships at about 2500 C, indicating that incipient melting may have taken place in the molybdenum rod used as a heat source. Because of the potentially higher useful temperature range and slightly better thermoelectric properties, rhenium-tungsten thermocouples may be of somewhat greater interest than rhenium-molybdenum couples. Either couple can be used only in a neutral or reducing atmosphere.

TABLE IV.—SUMMARY OF THERMOELECTRIC DATA FOR RHENIUM-TUNGSTEN AND RHENIUM-MOLYBDENUM THERMOCOUPLES.

| | Rhenium-Tungsten | | Rhenium-Molybdenum | |
|---|---|---|---|---|
| | 870 to 1720 C | 1720 to 2400 C | 870 to 1500 C | 1500 to 2600 C |
| Thermoelectric force, mv... | $E = 0.015\ T - 5.4$ | $E = 0.0093\ T + 4.4$ | $E = 23.9 - \frac{1250}{T}$ | $E = 0.0055\ T + 7.3$ |
| Thermoelectric power, mv per deg Cent......... | $\frac{dE}{dT} = 0.015$ | $\frac{dE}{dT} = 0.0093$ | $\frac{dE}{dT} = \frac{1250}{T^2}$ | $\frac{dE}{dT} = 0.0055$ |

Although in this preliminary study the reproducibility of the thermoelectric properties have not been tested, it is clear that the rhenium-tungsten and rhenium-molybdenum couples show more promise as very high temperature couples than the tungsten-molybdenum couple investigated recently (**10**). The latter couple generates about 2 mv at 1600 C as against 16 to 18 mv for the present couples at the same temperature. Rhenium-molybdenum and rhenium-tungsten alloys recently reported by British workers (**9**) are thought to have very interesting physical and mechanical properties, and may also be of interest for thermoelectric applications. We plan to establish the thermal stability behavior of the rhenium-tungsten and rhenium-molybdenum couples at a later date.

## RHENIUM-PLATINUM ALLOYS *versus* PLATINUM

Alloys of platinum - 1 and 2 per cent rhenium were prepared by arc melting and fabricated to fine wire as described in *Electrical Resistivity Studies*. The thermal electropotential of these two alloys were measured against platinum.

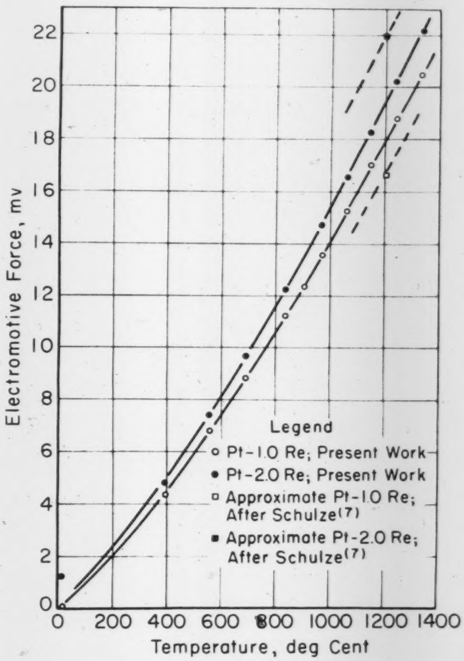

FIG. 4.—Thermoelectric Potential Data for Platinum-1.0 Rhenium and Platinum-2.0 Rhenium *versus* Platinum.

The thermocouples were placed in a protection tube in a molybdenum-wound tube furnace containing a reducing atmosphere. Two platinum *versus* platinum - 10 per cent rhodium couples were also included. The beads of all four couples were in small temperature-equalizing block of molybdenum. The thermoelectric potential data obtained are shown in Fig. 4 along with those of Schulze ((7).

Schulze found a greater difference in potential between the platinum *versus* platinum - 1 per cent rhenium and the platinum *versus* platinum - 2 per cent rhenium than was found in this work. No chemical analysis was obtained of the platinum-rhenium wires used in this work; Schulze also reported no analysis. It is possible that alloy composition may account for variations between the present data and those of Schulze.

*Acknowledgments:*

The authors wish to express their appreciation to the Aeronautical Research Laboratory of the Wright Air Development Center for financial support of this work. In addition, thanks are due the Kennecott Copper Corp., who supplied rhenium without charge.

The assistance of Bruce G. Ross and Wilbur W. Kleinschmidt, who conducted many of the actual measurements, is also greatly appreciated.

## REFERENCES

(1) C. T. Sims, C. M. Craighead, and R. I. Jaffee, "Physical Properties of Rhenium," *Journal of Metals,* Vol. 7, pp. 168–179 (1955).

(2) Battelle Memorial Institute, *WADC TR 54-371,* June, 1954.

(3) J. E. Hughes, "The Alloy System Rhenium-Carbon," Associated Electrical Industries, Ltd., *Research Report A497* (Nov., 1955).

(4) C. Agte, B. Alterthum, K. Becker, G. Heyne, and K. Moers, "Physical and Chemical Properties of Rhenium," *Zeitschrift für anorgonische u. allgemine Chemie,* Vol. 196, pp. 129–159 (1931).

(5) W. Meissner, and B. Voigt, "Measurements with the Aid of Volatile Helium; Part XI: Resistance of Rhenium Metal at Low Temperatures," *Annale des Physik,* Vol. 7, p. 892 (1930).

(6) C. T. Sims, and R. I. Jaffee, "Further Studies of the Properties of Rhenium Metal," *Journal of Metals,* Vol. 8, p. 913 (1956).

(7) A. Schulze, "Thermo-Elements at High Temperatures," *Zeitschrift des Vereines deutscher Ingenieure,* Vol. 77, pp. 1241–42 (1933).

(8) R. Vines, and E. Wise, *The Platinum Metals and Their Alloys,* The International Nickel Co., Inc., New York, N. Y. (1941).

(9) G. A. Geach, and J. E. Hughes, "The Alloys of Rhenium with Molybdenum and with Tungsten and Having Good High-Temperature Properties," Associated Electrical Industries, Ltd., *Research Report A479,* Sept., 1955.

(10) H. Greenaway, S. Johnston, and M. McQuillan, "High-Temperature Thermal Analysis Using the Tungsten-Molybdenum Thermocouple," *Industrial Heating,* pp. 2270–2280, Dec., 1952.

## DISCUSSION

Mr. W. L. Fink.[1]—When the thermal emf curves were plotted, they showed a break at recrystallization temperature. Now, if the specimens were cooled and the emf remeasured at the lower temperature, does the second value fall on the prolongation of the upper part of the curve?

Mr. C. T. Sims (*author*).—We were not able to conduct the experiment you discuss. However, we have authorization to continue and expand the work, and we plan to confirm this data. We will also include experiments on emf stability after thermal exposure, which should provide information about recrystallization effects.

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[1] Chief, Physical Metallurgy Division, Alcoa Research Laboratories, Aluminum Company of America, New Kensington, Pa.

# MECHANICAL BEHAVIOR OF CEMENTED CARBIDES*

By R. P. Felgar and J. D. Lubahn[1]

## Synopsis

The elastic, plastic, and fracture properties of various grades of Carboloy cemented carbide were determined by tension, compression, and torsion tests. The following properties were measured: modulus of elasticity, Poisson's ratio, proportional limit, yield strength, strain hardening exponent, ductility, and fracture strength.

In most respects, cemented carbide has a behavior which is qualitatively similar to that of metals: its plastic behavior conforms to the laws of classical plasticity; its ductility decreases with increasing hardness and increasing hydrostatic tension component; it exhibits a pronounced Bauschinger effect, though not quite so much so as metals; and it develops considerable hysteresis after some plastic flow. Cemented carbide is harder than ultra-high-strength steels and hardened tool steels because of its very high strain hardening rate; these steels have proportional limit values at least as high as cemented carbide with 6 per cent binder, and much higher than carbides with 15 per cent binder, but the strain hardening exponents of these steels are so much lower than those of the cemented carbides that steels do not have nearly as high a yield strength and hardness, particularly as compared to the 6 per cent cobalt carbides.

The properties of cemented carbide, particularly proportional limit, tensile strength, and ductility, were found to be much more consistent than in other investigations. Such consistency can only be obtained for such hard, brittle materials by careful control of the testing and analysis procedures, the accurate measurement of the slope of the elastic modulus line (to which the proportional limit is very sensitive) and the elimination of eccentricity of loading.

The properties of the carbides were found to vary with cobalt binder in the same manner as reported by previous investigators. At a given cobalt content, the yield strength increased with decreasing grain size and the ductility decreased with decreasing grain size.

This investigation was undertaken in order to explore the mechanical behavior of Carboloy cemented carbide[2] in somewhat more detail than previously. The use of carbides for dies and tools often involves such high stresses as to result in plastic flow, and certain applications

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* Presented at the Sixieth Annual Meeting of the Society, June 16–21, 1957.

[1] Mechanical Engineering Laboratory, General Engineering Laboratory, General Electric Co., Schenectady, N. Y.

[2] General Electric trade mark for metal compositions of tungsten carbide with cobalt binder.

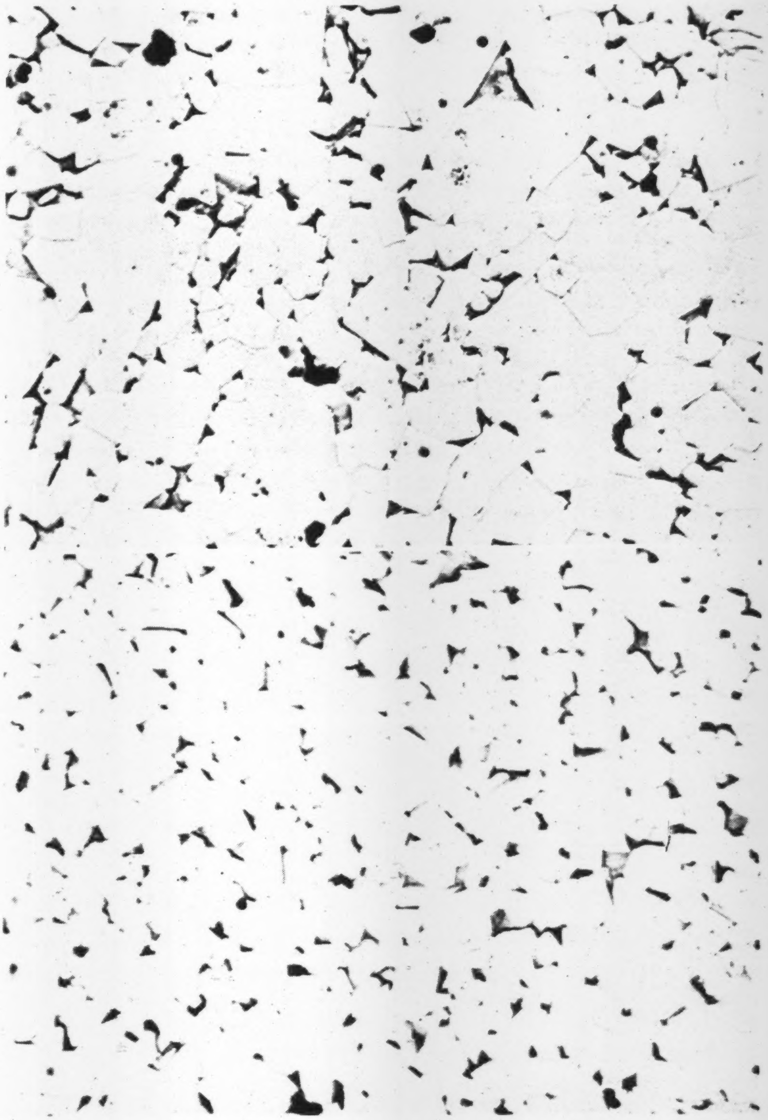
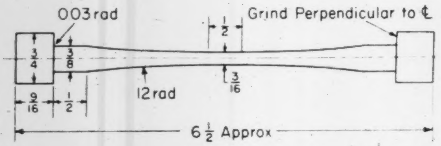


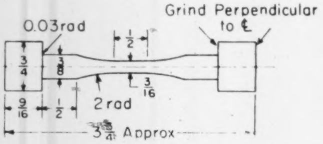
FIG. 1.—Electron Micrographs of Grade 44A (upper) and 883 (lower) (× 5000).

cause reversal of the direction of loading. Consequently it has become desirable to be able to handle stress analysis problems in the plastic region and for reversed loading. To this end. tension, torsion, and compression tests were made to obtain accurate data of the standard variety extending well into the plastic range, and also to explore in a preliminary manner the effects of a complex stress state and a reversed loading history on the plastic flow behavior. Also, an effort was made to develop better testing techniques, which would yield more reliable values of the properties.

Three grades of Carboloy were investigated, 55A, with 13 per cent cobalt, and 44A and 883, both with 6 per cent cobalt, but with different grain size distribution (Fig. 1). Some data on these, or similar materials, has been given previously by Engle (**1**),[3] Ballhausen (**2**), and Johnson (**3**).



(*a*) Tension and Torsion Test Specimens.



(*b*) Compression-Tension Test Specimen.

FIG. 2.—Test Specimens Used in the Investigation.

## EQUIPMENT AND PROCEDURE

### *Compression Tests*:

Specimens to be tested to failure were right circular cylinders $\frac{3}{8}$ in. in diameter and 1 in. high with the ends ground and lapped normal to the cylindrical surface. Figure 2 shows the type of specimen used for tests where compression is to be followed by tension.

Both types of compression specimens (and tension specimens too) were supplied near mid-length with three axial A-7-4- SR-4 gages spaced 90 deg apart and with one or two A-19 SR-4 gages in the transverse direction. Comparative readings on the three axial gages in the elastic region served to determine the eccentricity of loading. The transverse gages were used to obtain Poisson's ratio.

The load was applied through a spherical seat and polished carbide blocks. The load was centered by manually adjusting the spherical seat until the strain indicated by all axial gages were equal. If there was an appreciable initial eccentricity or an increase in eccentricity during the test, the edge of the loading head was tapped with a hammer until all gages indicated essentially the same strain. To show that the test results were not affected by the jarring of the head and changing the tilt of the head during the test, one specimen was unloaded after the tilting operation to check the zero reading. Figure 3 shows that the zero values were unaffected and that the only effect of the tilting was to remove the bending component. Eccentricity values in all compression tests were less than 1 per cent of the diameter of the specimen (average stress less than 8 per cent below maximum). In both compression and tension tests, centerline strain was obtained by averaging opposite surface strain.

One specimen was loaded in increments, and in the plastic region some creep occurred at each load level where the load was held constant. However, the creep was always a small fraction of the entire plastic strain, and so incremental loading gave the same result as continuous loading, within the limit of the scattering among specimens.

A special compression specimen of grade 55A was used to determine the effect of end constraint. This specimen was 0.27 in. in diameter and 0.92 in. high and therefore had a slightly higher height-to-diameter ratio (3.4) than the value of 2.7 for the standard specimens. The elastic modulus was found to be $79 \times 10^6$ psi at mid-height (1.7 diameters from the end), $89 \times 10^6$ psi at 1.25 diameters from the end, and $99 \times 10^6$ psi at 0.8 diameters from the end, thus indicating that some effects of end restraint still exist as far as 1.25 diameters from the end. Consequently specimens with a height-to-diameter ratio of 2.5 would not be expected to be free of end restraint even at mid-height. Although this ratio is only slightly higher (2.7) for the standard specimens, they were apparently free from end effects at mid-height, for the modulus values ($80 \times 10^6$ and $78 \times 10^6$ psi) were essentially the same as for the button-head specimens ($79 \times 10^6$ and $78 \times 10^6$), which are clearly free of end restraint.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 788.

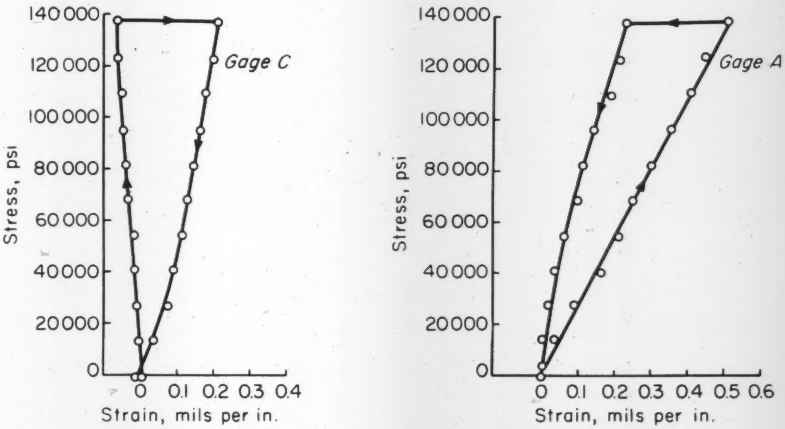

FIG. 3.—Effect of Tilting Compression Head While Specimen is under Load.

Two grade 55A compression specimens of the type shown in Fig. 2 were used to investigate the Bauschinger effect in Carboloy cemented carbide (a lowered elastic limit in tension because of plastic precompression). These were precompressed to 0.2 per cent plastic strain and then tested to failure in tension.

*Tension Tests*:

The specimens (Fig. 2) were provided with a large radius adjoining the test section, in order to minimize the stress concentration factor (1.005 for the 12-in. radius used). These were tested in a concentric loading fixture (see reference (**4**)) which gave eccentricity values less than 0.002 in. for all tests except one, where the loading surfaces under the button beads were not quite square with the specimen axis.

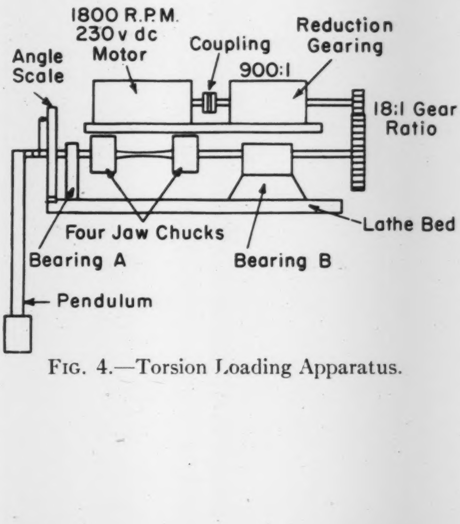

FIG. 4.—Torsion Loading Apparatus.

*Torsion Tests:*

Torsion tests were made to obtain a different stress state than the uniaxiality of tension and compression tests. The specimens are shown in Fig. 2 and the equipment in Fig. 4.

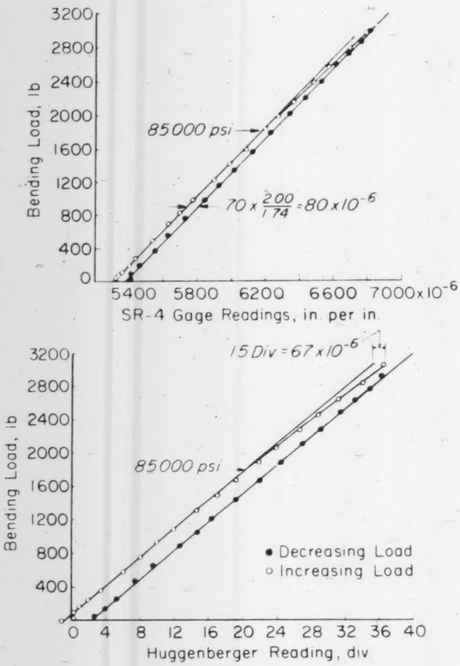

FIG. 5.—Comparison of SR-4 Gage Readings and Huggenberger Readings in Bending Grade 55B.

It was necessary to correct the torque measurements for friction in bearing A. The initial friction torque was measured by displacing the pendulum counterclockwise and reading the angle at equilibrium, then displacing the pendulum clockwise and again reading the angle. Considering also the location of the center of gravity and the weight of the pendulum, the initial friction torque was then computed to be about 5 in-lb. However, it was found during the running of the tests that the friction torque was larger, probably because the placing of the specimen in the chucks increased the transverse force on the bearing. To obtain the friction torque during testing, each specimen was loaded in the elastic region, unloaded, and then loaded to failure. The friction torque was calculated from the moment difference between the loading line and the average line between the loading and unloading curves (see Fig. 9). The friction torque obtained in this manner was found to be 15 to 19 in-lb. in the various tests.

Four Baldwin-Southwark type A-7-4 strain gages were placed on each specimen at approximately 45 deg to the axis of the specimen. The actual direction of the sensitive axis of each gage was measured by means of a machinist's microscope. These angles varied from 24 to 59 deg.

The data from the four strain gages of the torsion tests were used with Mohr's circle to determine four values of the principal strain and four values of the maximum shearing strain and these four values were averaged. The modulus of elasticity in shear was computed from the torque divided by the polar section modulus and the shearing strain. For comparison with Young's modulus, the shear modulus, $G$, was converted to Young's modulus, $E$, using Poisson's ratio ($\mu = 0.26$) from the uniaxial tests and the equation

$$G = \frac{E}{2(1 + \mu)}$$

*Reliability of Strain Measurements:*

To establish that SR-4 gages measure strain faithfully on cemented carbide, a 0.504 in. square bar of cemented carbide 55B (16 per cent cobalt binder) was tested in four point bending with an

A-8 SR-4 gage and a Huggenberger gage measuring tangential strain. The results, Fig. 5, show by both measuring methods that plastic flow begins at about 1800 lb. (85,000 psi) and that about 75 × $10^{-6}$ plastic strain had occurred at 3000 lb (140,000 psi). The elastic slopes agreed for loading and unloading, and corresponded to a modulus of 89,000,000 psi for the Huggenberger gages and 84,000,000 psi for the SR-4 gage. Thus, the two gages indicated essentially the same behavior, the difference between gage readings being about 6 per cent.

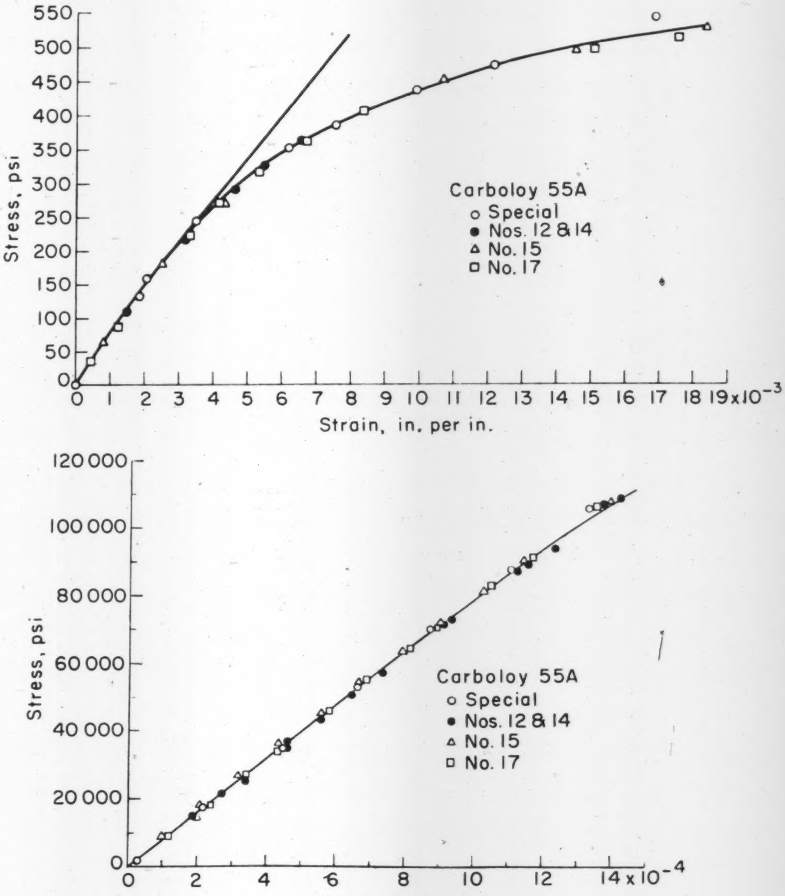

FIG. 6.—Compression-Test Diagram for Grade 55A.
Lower diagram is a magnification of the initial portion of the upper diagram.

## TEST RESULTS

The stress-strain curves for the tension and compression tests are shown in Figs. 6 to 8, and a typical torque-strain curve for one gage of a torsion test is shown in Fig. 9. The scattering of measured values about the trend line and the variations from specimen to

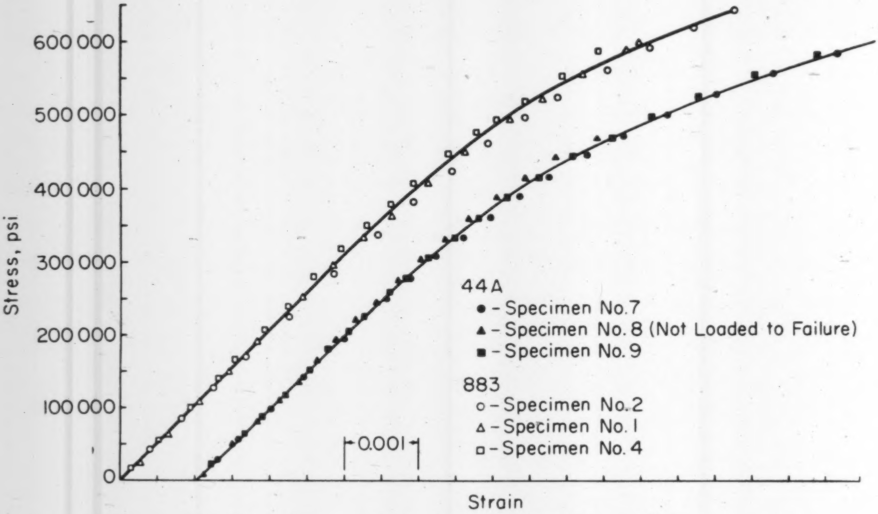

FIG. 7.—Compression Tests on Grades 44A and 883.

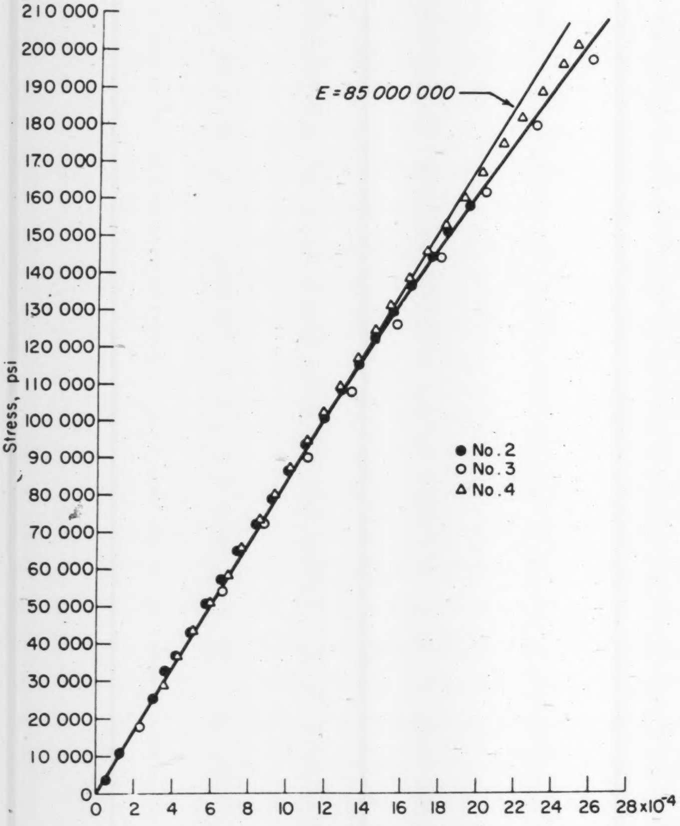

FIG. 8.—Tension-Test Diagram for Grade 55A.

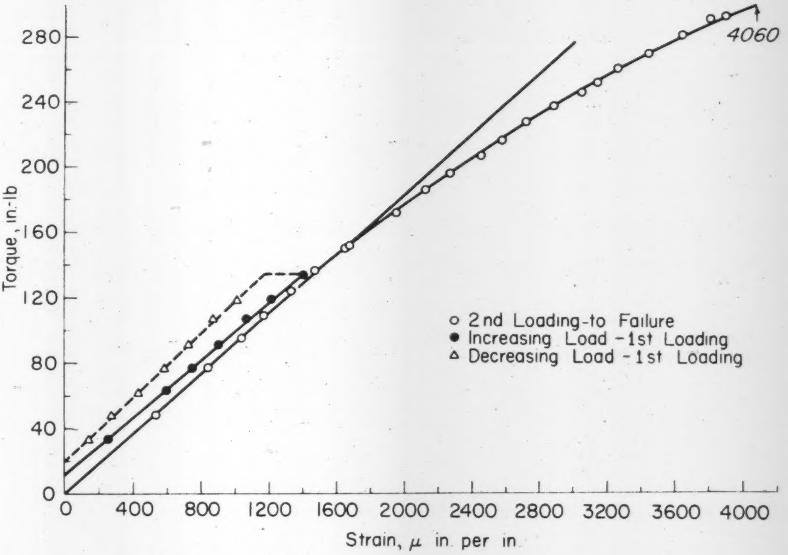

FIG. 9.—Torque-Strain Diagram for Grade 55A.

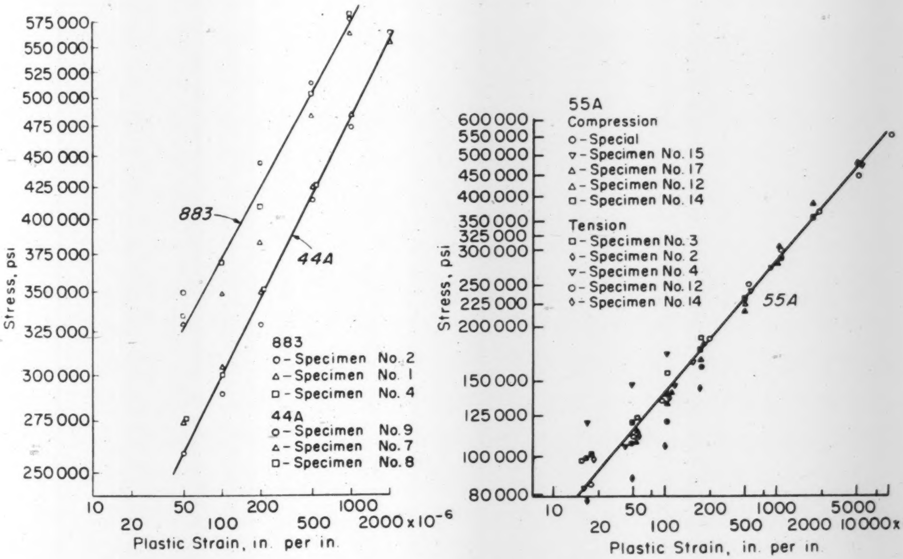

FIG. 10.—Stress *versus* Plastic Strain for Carboloy Cemented Carbide.

TABLE I.—MECHANICAL PROPERTIES OF CERMETS OF TUNGSTEN CARBIDE WITH COBALT BINDER.

| | 5.7 per cent Cobalt Ball-hausen | 6 per cent Cobalt 44A | 6 per cent Cobalt 883 | 6 per cent Cobalt | | | 6 per cent Cobalt Engle | 13 per cent Cobalt | | | 13 per cent Cobalt Engle | per cent Cobalt Ball-hausen | 15 per cent Cobalt | | | 16 per cent Cobalt 55B |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Johnson | | | | 55A | | | | | Johnson | | | |
| | Comp. | Comp. | Comp. | Tension | Comp. | Shear | Comp. | Tension | Comp. | Shear | Comp. | Comp. | Tension | Comp. | Shear | Bend |
| Elastic Properties: | | | | | | | | | | | | | | | | |
| Modulus of elasticity, $10^6$ psi | 88 | 100<br>105<br>101 | 100<br>108<br>107 | 90<br>95 | 91<br>91<br>89<br>88 | 92<br>89 | 88 | 87<br>82<br>85 | 79<br>80<br>78<br>79<br>78 | 83<br>103<br>88 | 80 | 77 | 77<br>59 | 76<br>76 | 74<br>74 | 86 |
| Average | | 102 | 105 | 92 | 90 | 90 | | 85 | 79 | 91 | | | 68 | 76 | 74 | |
| Poisson's ratio | | 0.23<br>0.28<br>0.27 | 0.26<br>0.32<br>0.26 | | 0.21<br>0.22 | | | 0.28<br>0.25<br>0.27 | 0.26<br>0.29<br>0.26<br>0.24<br>0.23 | | | | | 0.22<br>0.23 | | |
| Average | | 0.26 | 0.28 | | 0.22 | | | 0.27 | 0.25 | | | | | 0.22 | | |
| Plastic Properties: | | | | | | | | | | | | | | | | |
| Proportional limit, 1000 psi | | 210<br>210<br>210 | 280<br>300<br>280 | 70 | 280<br>305 | | 600 | 83<br>85<br>96 | 91<br>73<br>72<br>78<br>78 | 92<br>90<br>90 | 525 | | 45<br>10 | 165<br>170 | 60<br>60 | 85 |
| Average | | 210 | 285 | 70 | 290 | | | 88 | 78 | 91 | | | 30 | 170 | 60 | |
| 0.2 per cent yield strength, 1000 psi | | 555<br>565 | | | 540 | | | | 365<br>355<br>360<br>345<br>340 | | | | | 365<br>370 | | |
| Average | | 560 | | | 540 | | | | 355 | | | | | 370 | | |
| Strain Hardening Exponent | | 0.21 | 0.19 | | | | | 0.30 | 0.30 | | | | | | | |

| | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Fracture Properties: | | | | | | | | | | | | | | | | |
| Fracture stress, 1000 psi | | 675<br>655 | 650<br>670<br>640 | | | | | 200<br>225<br>210 | 570<br>550<br>560 | | | | | | | |
| Average | | 665 | 650 | | | | | 210 | 560 | | | | | | | |
| Average } Fracture stress, 1000 psi<br>Nominal } | 825 | 645<br>650 | 600<br>640<br>600 | 80<br>80 | 540<br>525<br>510<br>535 | 60<br>100 | 750 | 160<br>210<br>200 | 550<br>530<br>520 | 215<br>220<br>240 | 625 | 590 | 120<br>65 | 510<br>510 | 130<br>135 | 280 |
| Average | | 650 | 615 | 80 | 530 | 80 | | 190 | 530 | 225 | | | 90 | 510 | 130 | |
| Fracture strain, per cent | | 0.42<br>0.45 | 0.14<br>0.18<br>0.15 | | | | | 0.020<br>0.037<br>0.019 | 1.0<br>1.2<br>1.2 | | | | | | | |
| Average | | 0.44 | 0.16 | | | | | 0.025 | 1.1 | | | | | | | |
| Average } Fracture strain, per cent<br>Nominal } | | 0.42<br>0.43 | 0.13<br>0.17<br>0.14 | 0.004<br>0.000 | 0.17<br>0.19 | | | 0.016<br>0.035<br>0.018 | 1.0<br>1.2<br>1.1 | 0.15<br>0.16<br>0.19 | | | 0.014<br>0.022 | 1.3<br>1.5 | 0.04<br>0.05 | 0.05 |
| Average | | 0.42 | 0.15 | 0.002 | 0.18 | | | 0.023 | 1.1 | 0.17 | | | 0.018 | 1.4 | 0.05 | |

specimen which are evident in Figs. 6 to 8 are also characteristic of the torsion tests. In all types of tests on all materials the behavior was similar: deviation from the initial straight-line portion occurred quite early and very gradually, and the stress-strain curve was quite steep even at stresses that were several times higher than the elastic limit.

The plastic behavior can be seen more clearly by plotting only the plastic part of the strain against the stress on log-log coordinates (Fig. 10). In these graphs, what appears as large scattering at small plastic strains arises from small uncertainties in the slope of the elastic line. A small error in the elastic slope results in a small error in estimating the deviation from the elastic line (plastic strain), which is magnified by the log scale of Fig. 10. The slope of the log stress-log plastic strain curve is a measure of the strain hardening rate and is called the "strain hardening exponent."

From the various types of tests made, standard properties characterizing the elastic, plastic, and fracturing behavior were obtained. The elastic behavior is characterized by the elastic modulus and Poisson's ratio; the plastic behavior is characterized by the elastic limit, the yield strength, and the strain hardening exponent; and the fracturing behavior is characterized by the fracture stress and ductility. Table I shows values of these properties derived from the tests of this investigation as well as from previous investigations. The results from this investigation show much less scatter than is usually observed in such hard, brittle materials.

Young's modulus for cemented carbide ($80 \times 10^6$ to $105 \times 10^6$ psi) is essentially the same for tension, compression, and shear tests and is lower for the higher

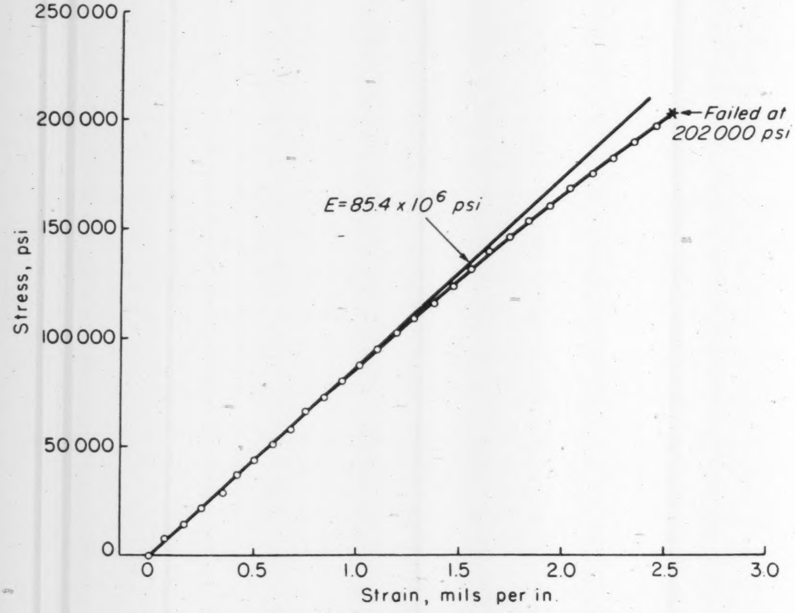

FIG. 11.—Tension Test Diagram for Grade 55A.

cobalt contents. These trends are also evident in the data from previous investigators, but for each composition the values are somewhat lower than those from the present work. Grain size is not given in the previous investigations, and so comparisons cannot be made for comparable grain sizes; on the other hand, comparison of the present data for two grain sizes (grades 44A and 883) indicates little effect of grain size on elastic properties or ultimate compressive strength.

Poisson's ratio is about 0.26, independently of composition or grain size. Johnson's data show the same result, but his values are a little lower (0.22).

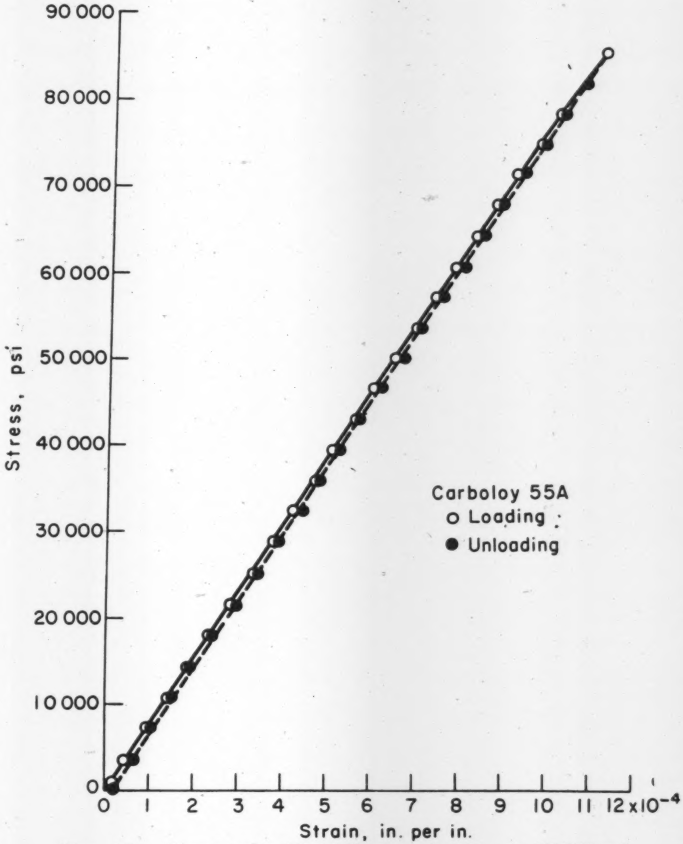

FIG. 12.—Compression Test Diagram Loading to 86,000 psi.

The present work indicates that the proportional limit is the same in tension, compression, or shear, and that decreasing cobalt content or decreasing grain size increases the proportional limit. The proportional limit is quite low compared to the yield strength: 22 per cent of the yield strength for grade 55A and 38 per cent of the yield strength for grade 44A. Proportional limit values from previous work scatter widely: for cermets with 13 to 15 per cent cobalt the data show values ranging from as little as 10,000 psi (Johnson-tension) to as much as 525,000 (Engle-compression).

The discrepancies in elastic limit described above are probably due to uncertainties in fairing the stress-strain curves. In carbide the plastic line deviates so gradually from the elastic line that the point of first deviation is quite uncertain and very sensitive to small variations in the slope of the straight line that is used to fair the points in the initial region. As shown in Fig. 11, where the usual testing procedure has been employed, the elastic limit may be selected anywhere between 70,000 and 130,000 psi (±30 per cent) depending on how the elastic modulus line is drawn. When the specimen is unloaded from just beyond the elastic limit, the slope of the straight unloading line defines more accurately the loading line. The correspondingly greater accuracy of the elastic limit is indicated by Fig. 12, where the conceivable range of elastic limit values from 75,000 to 80,000 psi represents an uncertainty of only 3 per cent at the greatest. This procedure of unloading from slightly above the elastic limit was used to determine the elastic limit in most cases.

Values of proportional limit obtained by estimating the departure from linearity of the stress-strain curve are in agreement with conclusions drawn from log stress - log plastic strain curves (Fig. 10). The estimated proportional limit corresponds in all cases to a stress for $20 \times 10^{-6}$ plastic strain. This is about the limit of strain sensitivity and therefore the natural criterion of "zero" plastic strain or "elastic limit."

This yield strength varies with cobalt content and grain size in the same way as proportional limit. The ductility is not high enough in grade 883 to obtain a 0.2 per cent yield strength value, even in a compression test, but the relative yield strengths for different grain sizes can be observed for the 0.1 per cent yield strength (Fig. 10): 480,000 psi for the coarse-grained grade 44A as compared with 570,000 psi for the fine-grained grade 883. The yield strength values obtained in this investigation agree well with those reported by Johnson.

The strain hardening exponents for the grades of Carboloy cemented carbide tested are higher than for most steels. The value for 13 per cent-cobalt material is 0.30, while that of the 6 per cent-cobalt materials is close to 0.20. This means that the stress-strain curve does not rise quite so steeply (percentage-wise) for the harder carbides as for the softer grade, or, in other words, the ratio of 0.2 per cent yield strength to proportional limit is smaller. The very high hardness of cemented carbide is associated primarily with this high strain hardening exponent. Ultra-high-strength steels and tool steels have proportional limit values at least as high as carbide with 6 per cent cobalt binder and much higher than that of grade 55A, but the strain-hardening exponents of these steels are quite low. Consequently they do not have nearly as high a yield strength and hardness, particularly compared with the 6 per cent cobalt types.

In Table I, both the average (or nominal) and local fracture properties have been reported. In the case of the torsion tests, the nominal fracture strain is the offset from the elastic modulus line at fracture and the nominal fracture stress is the torque at fracture divided by the polar section modulus. The reporting of local values essentially constitutes a correction for eccentricity, and so a comparison of such values for duplicate tests gives a true measure of the reproducibility of the material. Table I shows that the material gives very good reproduciblity even in tension tests. Probably the high scattering that is usually encountered in such hard, brittle materials is caused by a failure to avoid, or correct for, eccentricity of loading.

Table I shows that the ductility is smallest in the tension test, largest in the compression test, and intermediate in the torsion test. These observations are in agreement with the oft-quoted generalization that ductility decreases with increasing hydrostatic component, or average principal stress

$$\left(\frac{S_1 + S_2 + S_3}{3}\right).$$

The variations in ductility with type of test are reflected in the fracture stress: the fracture stress is smallest when the ductility is smallest.

In general, the higher the yield strength and proportional limit, the lower is the ductility in a given type of test. The ultimate strength depends on both the yield strength and ductility; the yield strength is a measure of how high the stress-strain curve is, and the ductility is a measure of how far out on this curve the fracture point lies. The ultimate strength may be either higher or lower for one metal than another, depending on the relative importance of the yield strength effects and ductility effects. Apparently the greatest ultimate strength occurs in material of intermediate hardness (grade 44A). Grade 55A has more ductility but so much lower yield strength that the ultimate strength is less than that of grade 44A. Grade 883 has a very high

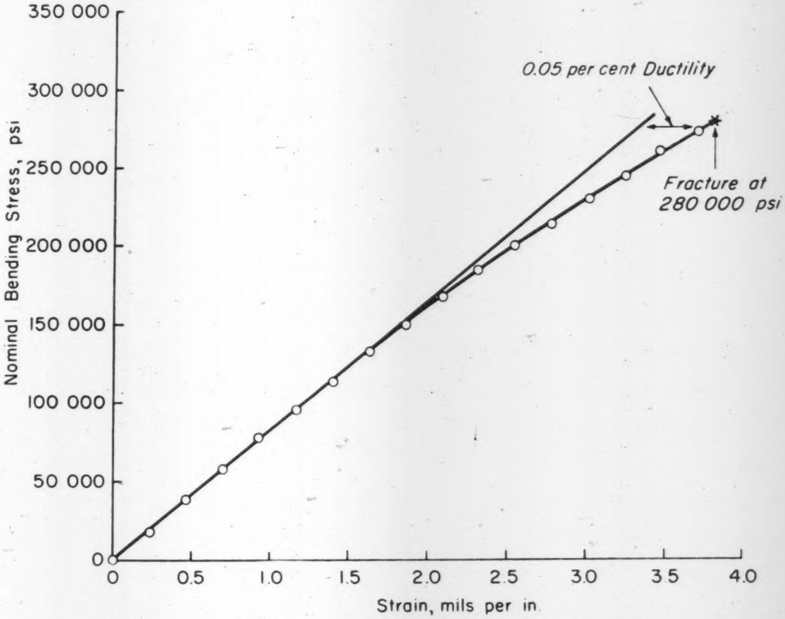

Fig. 13.—Complete Bending Load Diagram for Grade 55B.

yield strength, but so much lower ductility that the ultimate strength is less than that of 44A. Johnson's data also show a similar trend: the proportional limit increases with decreasing cobalt content but the ductility decreases, and consequently the ultimate strength passes through a maximum at about 10 per cent cobalt.

Although intended for showing the reliability of the gages, the bend test on grade 55B (Fig. 13) also provides some additional information on the trends with cobalt content. The bend test results in the same stress state as the tension test (uniaxial tension), and so can be compared with the tensile data on grade 55A, which contains slightly less cobalt (13 per cent, as compared with 16 per cent for grade 55B). The larger cobalt content results in more ductility and a higher fracture stress, as observed previously for the compression tests. There was essentially no difference in elastic or plastic properties between the 13 and 16 per cent cobalt grades.

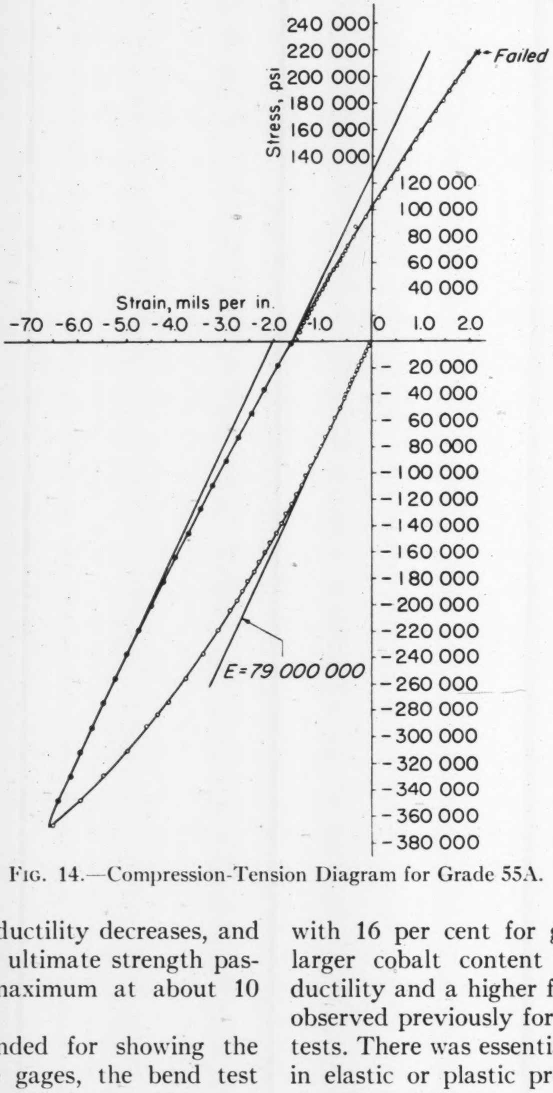

FIG. 14.—Compression-Tension Diagram for Grade 55A.

Figure 14 shows a typical result of the two reversed loading tests for investigating the Bauschinger effect in carbides by observing how early the compressive stress-strain curve deviated from linearity after plastic precompression. After about 0.2 per cent plastic strain in compression, the line for unloading and reversed loading showed considerable curvature, indicating that anelasticity (hysteresis) had been caused by that much plastic strain. (Note from Fig. 12 that small amounts of plastic strain do not introduce significant *anelastic* effects.) These hysteresis effects make it very difficult to observe the magnitude of the Bauschinger effect, because they result in deviation from linearity even in the absence of the Bauschinger effect. Consequently the magnitude of the Bauschinger effect can only be revealed by first subtracting the anelastic strain and thereby reveal the onset of plastic flow as the deviation from the true linear elastic behavior. The anelastic strain, $e_a$, was determined (5) from the curvature of the unloading line using a method (6) that involves the concept of a relaxation spectrum and can be expressed as a function of stress, $S$, and time, $t$, in seconds by the following equation:

$$e_a = \frac{S}{E}\,[0.079(1 - e^{-t/50}) + 0.318(1' - e^{-t/5000})]$$

(This anelastic strain function is not very accurate, because the need for anelastic correction was not anticipated, and consequently the unloading history was known only approximately.) The equation indicates that most of the anelastic strain is characteristized by the rather long time constant of more than 1 hr (5000 sec.).

Figure 15 shows the result of subtracting the anelastic strain from the measured stress-strain curve in tension. The anelastic strain was determined as previously, using the anelastic strain function derived above and the (approximately) measured tensile loading

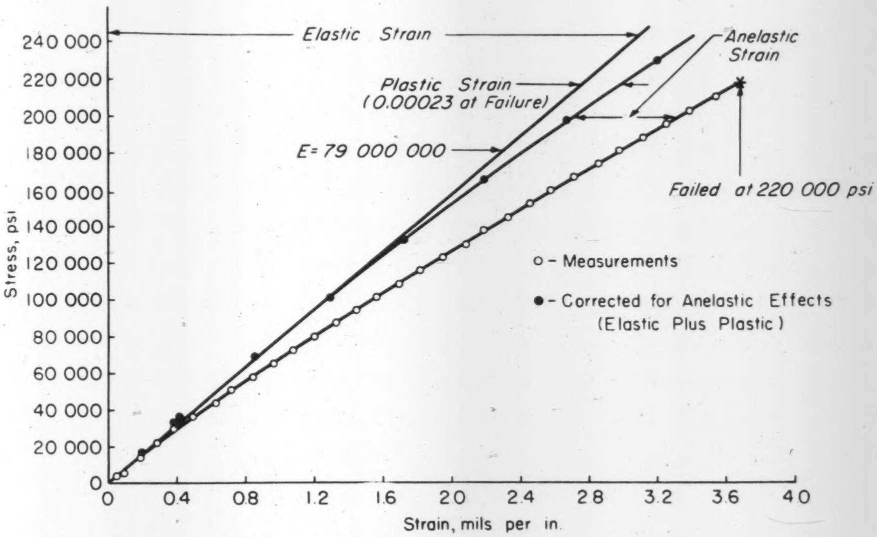

FIG. 15.—Plastic Behavior of Grade 55A in Tension after Plastic Precompression.

history. The figure shows that the elastic-plus-plastic behavior is linear up to 100,000 psi (100,000 psi elastic limit) with a modulus of 79 × $10^6$ psi, which agrees with the values from other tests (Table I), with subsequent gradual deviation amounting eventually to 230 × $10^{-6}$ plastic strain at a stress of 220,000 psi (0.023 per cent yield strength equals 220,000 psi). These values of elastic limit and yield strength are slightly higher than values of 90,000 psi and 180,000 psi (see Table I and Fig. 10) for virgin specimens, and so it may be concluded that the onset of plastic flow in tension is not hastened by precompression, as is observed in most materials (the Bauschinger effect). However, plastic flow *does* occur much sooner than one would expect if the full strain hardening from the precompression had prevailed; in this case, one would expect the elastic limit to be equal to the 0.2 per cent yield strength, which is 355,000 psi according to Fig. 10. In other words, there is a pronounced Bauschinger, effect in Carboloy cemented carbide but not to quite such an extent as in metals.

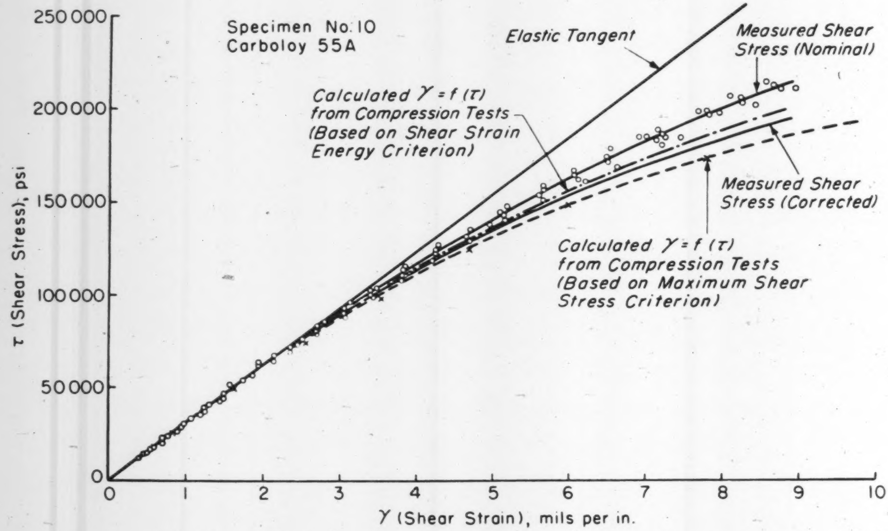

FIG. 16.—Comparison of Measured and Calculated Shear Stress-Shear Strain Curves.

The tensile fracture properties are not affected by plastic precompression (see Table I).

The stress-strain relationship in torsion was computed and compared with that obtained in the compression tests. Figure 16 shows the nominal shear stress-shear strain curve obtained from Fig. 9 by dividing the torque by the shear section modulus and converting normal strain to shear strain. This procedure assumes that the shear stress is a linear function of radius. A corrected curve obtained by a method suggested by Nadai (7) is also shown. This curve is based on the shear strain being a linear function of radius and shear stress being a unique function of shear strain.

The shear stress-shear strain curves are also calculated from the compression tests data: two calculated curves are shown, one is based on the shear strain energy flow criterion (Huber-V. Mises-

Henckey) and the other on the assumption that the maximum shear strain is a unique function of maximum shear stress (Guests's law). It is interesting to note that the measured values lie between the two calculated curves but closer to the distortion energy one. This behavior agrees with other observations on steel and aluminum, and consequently it can be concluded that the same methods used for the plastic analysis of metals are valid for cemented carbide.

## CONCLUSIONS

1. The eccentricity in compression testing can be adjusted to less than 1 per cent of the specimen diameter before or during the test by tapping the spherical loading head.

2. In the elastic region there is no effect of end restraint at mid-height of compression specimens whose length-to-diameter ratio is greater than 2.7, but specimens with a length-to-diameter less than 2.5 would be expected to have an effect of end restraint at mid-height.

3. In all grades tested, the strain hardening rate was quite high (higher than most metals), resulting in yield strength values much higher than the elastic limit values.

4. The scattering in property values is much less than usually observed in hard, brittle materials. This is ascribed to the many precautions taken in the testing procedures.

5. Young's modulus is essentially the same in tension, compression, and shear tests, and is lower for higher cobalt contents. These observations confirm the findings of Johnson.

6. Poisson's ratio is 0.26, independently of grain size or composition. Johnson obtained 0.22.

7. Proportional limits and yield strengths are the same for tension, compression, or shear tests, and increase with decreasing cobalt content or decreasing grain size.

8. In this investigation, values of proportional limit are very consistent. Values from other investigations scatter over a range of fifty to one, probably because of relatively small errors in the slope of the elastic line.

9. Cemented carbide has a strain hardening exponent that is higher than that of most steels.

10. Ultra-high-strength steels and tool steels have proportional limit values at least as high as carbide with 6 per cent binder and much higher than that of grade 55A, but the strain hardening exponents of these steels are so much lower than that of cemented carbide that they do not have nearly as high a yield strength and hardness, particularly compared to the 6 per cent cobalt carbides.

11. The nominal values of fracture strength and ductility show supprisingly low scatter, probably because of the low eccentricity values. This scatter can be still further reduced by measuring the eccentricity and correcting for it.

12. As in metals, the fracture strength and ductility for a given grade decrease with increasing hydrostatic stress component.

13. The higher the yield strength, the lower is the ductility. Ultimate strength depends in a complex way on both yield strength and ductility.

14. After some plastic flow, cemented carbide exhibits a pronounced hysteresis effect which is characterized by a comparatively long time constant.

15. There is a pronounced Bauschinger effect in carbide, but not to quite such an extent as in metals.

16. The same methods used for the plastic analysis of metals are valid for cemented carbide. The energy-of-distortion criterion applies somewhat more accurately than the shear stress law.

*Acknowledgment*:

The authors are indebted to R. Plunkett, of the General Electric Co., General Engineering Laboratory, for the plasticity analysis of torsion test data, and to R. Dorr, of the General Electric Co., Metallurgical Products Department, for assistance in making the tests. The specimens were prepared by the Metallurgical Products Department, and the tests were carried out at the General Electric Co., Research Laboratory. The electron micrographs were prepared at the General Electric Co., Research Laboratory by the courtesy of E. R. Stover.

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## REFERENCES

(1) E. W. Engle, "Cemented Carbides," *Powder Metallurgy*, pp. 436–453. Papers presented at the 1940 and 1941 conference on Power Metallurgy held at Massachusetts Inst. of Technology, edited by John Wulff, Am. Soc. Metals (1942).

(2) C. Ballhausen, "Ein Ordnugssystem der Hartmetalle," *Pulvermetallurgie*, pp. 221–231. Papers delivered at the first Plansee seminar, "De Re Metallica," edited by F. Benesowsky, Springer, Berlin (1953).

(3) A. E. Johnson, "Mechanical Properties at Room Temperature of Four Cermets of Tungsten Carbide with Cobalt Binder," *Technical Note 3309*, Nat. Advisory Committee on Aeronautics, Washington, D C., Dec. 1954.

(4) G. Sachs, J. D. Lubahn, and L. J. Ebert, "Notch Tensile Test Characteristics of Heat Treated Low Alloy Steels," *Transactions*, Am. Soc. Metals, Vol. 33, p. 340 (1944).

(5) R. P. Felgar and J. D. Lubahn, "Material Properties of Carboloy Cemented Carbide, Grade 55A," *Report No. R56GL42*, General Electric Co., General Engineering Lab. Jan. 12, 1956.

(6) J. D. Lubahn, "The Role of Anelasticity in Creep, Tension, and Relaxation Behavior," *Transactions*, Am. Soc. Metals, Vol. 45, p. 787 (1953).

(7) A. Nadai, "Theory of Flow and Fracture of Solids," Vol. 1, McGraw-Hill, p. 347–350 (1950).

# DISCUSSION

MR. J. T. RICHARDS.[1]—The curves appear to be elastic well beyond the elastic limit cited. Was any attempt made to plot according to stress deviation, which would show considerably more sensitivity? It appears that the true elastic range extends beyond the 120,000-psi figure given.

We have also observed in our own tests the wave-like pattern of points around the modulus line, but we have yet to find a suitable explanation.

MR. J. D. LUBAHN (*author*).—We did not actually measure elastic limit proper, that is, by loading up to successively higher loads and then unloading to see whether there is plastic strain. In most of the tests, however, we did follow the procedure that I showed in one figure, namely, unloading after there was just a very slight amount of deviation from the straight line. In all cases it turned out that at that point there was a little bit of plastic strain, namely, that the unloading line was displaced from the loading line a little. Consequently, at least you can say that after we had exceeded the proportional limit, we had also exceeded the elastic limit. Whether they are exactly the same or not, I could not say.

Regarding the comment about periodicity of points about a line, I wonder whether this might not be an extraneous effect from reading. I remember having obtained the same result using a dial gage type of extensometer and discovering that I was reading the gage a little closer to the line when the pointer was near the line than when midway between lines, and getting a periodicity of one division. I can envision all sorts of similar errors. We did not notice this periodicity in the present tests.

MR. RICHARDS.—It would be very interesting to see the data plotted in the elastic region with suitable corrections for true stress and true strain. The curves would probably show much higher proportional limits. If that were the case, one would expect a slightly higher modulus in compression than in tension because of the greater area with increased loading.

MR. LUBAHN.—It would not make very much difference whether true or conventional stress were plotted. The modulus is so high that even though the proportional limit is fairly high, there still is not very much elastic strain at that point. Consequently, the cross-section is still essentially the same; it differs by only roughly 0.2 per cent. This means that the true stress would be different from the nominal stress by only 0.2 per cent, and we usually are not concerned with differences that small.

MR. S. L. HOYT.[2]—I should like to compliment the authors for presenting something that is new but, in view of our early experience, would be unpredictable.

I remember using a little peening hammer to test the strength of Carboloy. That gave me a good idea of the strength, but I missed the phenomenon of plastic deformation. The authors do not mention transverse rupture strength. We developed that test at a rather early period because we did need a numerical value for the strength of Carboloy. We also measured the Rockwell hardness, A scale, and those three tests were our devices for determining the mechanical properties and the mechanical behavior of Carboloy cemented carbide. They led to a definite opinion about the brittleness of the material and I was quite shocked to hear of strain hardening, elastic limit, and plastic deformation. Mr. Emmons at Cleveland Twist Drill had developed a very delicate and precise torsion test for brittle materials like his twist drills, and so we prevailed upon him to test Carboloy. The tests confirmed our initial experience that these materials were brittle.

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[1] Chief Engineer, Penn Precision Products, Inc., Reading, Pa.

[2] Metallurgical Consultant, Columbus, Ohio.

There is a good question of how plastic deformation can occur in that kind of a material. Cobalt is present in very thin films, which cement the brittle carbide particles together, *en masse*, and must lose its bulk properties and behavior. Has the microscopic behavior of the test specimens been examined to explain how an ordinary brittle material can exhibit all these plastic phenomena? Is it slippage in the thin cement films or just what is it?

MR. LUBAHN.—Regarding the last question, we did not specifically attempt to find the mechanism of plastic flow, that is, whether it was by slip-bands, twinning, etc. There has been a lot of study in metals as to different plastic flow mechanisms, but very little in cemented carbides. However, we do know that things which are brittle under one set of conditions can be ductile under others. Bridgman certainly showed that very well when he superimposed high hydrostatic pressures. As a matter of fact, materials like marble and other stone and ceramic materials were found long ago to show ductility in compression, while they would not have shown any in tension. Presumably the mechanism that permits plastic flow in those materials also permits plastic flow in cemented carbides.

The General Electric Research Laboratory is doing some work on this subject of deformation mechanism, but I am not up to date on their latest concepts.

# EFFECT OF COLD ROLLING AND HEAT TREATMENT ON THE DIRECTIONAL PROPERTIES OF BERYLLIUM COPPER STRIP*

By John T. Richards[1] and Kiyoshi Murakawa[2]

## Synopsis

The effects of cold rolling, solution treating and precipitation hardening upon the directionality of beryllium copper strip were investigated. Tension, conductivity and bending tests were employed to measure directional characteristics. In addition, the influence of aging time and temperature were considered. Some of the results obtained cannot be adequately explained by existing theories of precipitation hardening.

The role of precipitation upon directionality has received little attention in the past. Most studies of precipitation hardening have followed property changes in a unidirectional manner only. In the current investigation, an attempt has been made to evaluate the influence of precipitation upon directional characteristics.

Although long recognized, the directional characteristics of strip and sheet materials are far from being fully appreciated. Variations in mechanical and physical properties with test direction will, of course, influence design and performance. Better known, perhaps, is the influence of directionality on bending and drawing of cold-rolled material. A familiar example is found in the "earing" of deep-drawn cups.

Unfortunately, directional effects vary widely among available metals and alloys. This necessitates a critical examination of each material. Although the properties of strip and sheet may well vary with the three principal directions, practical considerations generally limit investigations to longitudinal and transverse directions (or to intermediate angles), in view of the magnitude of thickness with regard to length and width.

In most cases, preferred orientation is produced, altered, or removed by either cold work or recrystallization resulting from annealing (and solution treating). In the case of cold work, directionality increases with increasing amounts of rolling; however, the role of plastic deformation may prove complex, since both lattice distortion and preferred orientation may be factors. Cook and co-

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Chief Engineer, Penn Precision Products, Inc., Reading, Pa.

[2] Professor, Institute of Science and Technology, Tokyo, Japan.

workers (**1**),[3] in studying the influence of cold deformation upon elastic modulus, have observed differences in lattice distortion resulting from the generation of mobile dislocations after small strains and coarser-scale structural discontinuities following larger strains.

Upon heating, the effects of cold work may be modified, so that further changes in directionality can be expected. Heating above the recrystallization temperature either eliminates preferred orientation or produces a new one. The choice between these two conditions is apparently governed by the degree of prior cold reduction. Consequently, annealing or solution treating may not remove directionality but may even increase it, particularly in the case of severe prior deformation.

Heating at temperatures below the recrystallization range not only reduces lattice distortion but may also cause minor changes in orientation. Farnham and O'Neill (**2**), and subsequently Kawasaki (**3**), noted changes of hardness and orientation in copper following exposure to temperatures too low to cause recrystallization.

Earlier tests (**4,5**) on heat-treatable beryllium copper strip have demonstrated the presence of marked directionality in spite of the relatively light cold-rolling reductions generally applied (temper rolling limited to 37 per cent reduction in thickness). Subsequent tests on beryllium copper wire have shown that certain combinations of cold drawing and solution treating can produce marked preferred orientation. For example, with wire previously cold drawn 84 per cent, solution treating for 1 hr at 1470 F increased the E modulus[4] 54 per cent from 16,050,000 psi to 24,640,000 psi (**6**), while the same treatment decreased the G modulus 10 per cent from 5,594,000 psi to 5,024,000 psi (**7**). These rather large changes suggest the presence of pronounced orientation.

For beryllium copper and other age-hardening alloys, directionality studies may be complicated by precipitation and related phenomena. New phases, stress relief, and local recrystallization in areas of high stress concentration are all factors to be considered. A further complication is found in the rate of precipitation, which probably is not uniform throughout the structure.

Consequently, an understanding of the age-hardening processes in beryllium copper is necessary to evaluate directional characteristics. Copper will dissolve about 2.1 per cent beryllium at temperatures in the solution-treating range of 1400 to 1525 F with decreased solubility at lower temperatures. Rapid cooling (quenching) from a temperature within this field of maximum solubility will preserve this condition, so that there exists at room temperature a super-saturated solid solution ($\alpha$-phase) with relatively high ductility and relatively low hardness, strength and conductivity. Since only about 0.25 per cent beryllium is soluble in copper at the usual aging range of 500 to 750 F, heating within this range will cause precipitation of $\gamma$-phase from the unstable solution. Beryllium in excess of the 2.1 per cent $\alpha$-phase solubility limit forms $\beta$, an extremely hard solid solution consisting of approximately 4 to 10 per cent beryllium, balance copper.

In spite of numerous investigations, the mechanics of the hardening phenomenon and the relationship between property changes and actual precipitation are not fully understood. Three recent studies (**8,9,10**) have found evidence of one or more transition phases[5] prior to the formation of equilibrium $\gamma$. The influence of these unstable phases upon various properties, unfortunately, has not been completely established. Several investigators (**8,12,13**) have called attention to the occurrence of platelike segregates known as Guinier-Preston zones, which may in part account for certain changes in hardness and conductivity.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 806.

[4] As used in this text, the word modulus without further qualification means Young's modulus (E modulus).

TABLE I.—TEST MATERIALS.

| | Lot A | Lot B | Lot C | | |
|---|---|---|---|---|---|
| Composition, per cent: | | | | | |
| Beryllium | 1.91 | 1.87 | 1.86 | | |
| Cobalt | 0.29 | 0.39 | 0.19 | | |
| Iron | 0.24 | 0.22 | 0.16 | | |
| Silicon | 0.14 | 0.08 | 0.07 | | |
| Aluminum | 0.06 | 0.03 | 0.02 | | |
| Tin | trace | trace | 0.01 | | |
| Nickel | 0.02 | 0.02 | 0.01 | | |
| Lead | 0.000 | 0.000 | 0.000 | | |
| Zinc | 0.00 | 0.00 | 0.03 | | |
| Copper | balance | balance | balance | | |
| Temper | A | ½ H | A | ¼ H | H |
| Thickness, in. | 0.0201 | 0.0218 | 0.0220 | 0.0183 | 0.0146 |
| Reduction, per cent | 0 | 21 | 0 | 17 | 34 |

The following picture is obtained from studies by Gruhl (**8**) and Guy (**13**). Formation of Guinier-Preston zones causes the initial increase in resistance noted at low aging temperatures, while subsequent development of $\gamma'$- and $\gamma$-phases causes a decrease in resistance. Guy not only has shown a definite relationship between the presence of grain boundary precipitate and the drop in resistance, but also attributes the increase in hardness to Guinier-Preston zones. On the basis of Gruhl's work, some of the changes observed by Guy may be due to the transition phase $\gamma'$ rather than Guinier-Preston zones. Furthermore, Gruhl has presented evidence indicating that aging above 615 F or prior cold work leads directly to equilibrium precipitate $\gamma$, while suppressing the formation of either Guinier-Preston zones or intermediate $\gamma'$-phase.

More recently Geisler (**9,14**) has presented a slightly different view. Three different precipitates ($\gamma''$, $\gamma'$, and $\gamma$) were identified upon aging at 570 F. The initial increase in resistance noted at low temperature is believed due to coherency strain which more than offsets the decrease in resistance accompanying depletion of the solid solution. Geisler (**9**) has calculated that the strain resulting from precipitate formation is sufficient to cause plastic deformation of the matrix. The increase in resistance is comparable to that caused by moderate cold working; however, at higher temperatures solid-solution decomposition causes a rapid decrease in resistance, masking the smaller and opposite effect of matrix strain. Geisler (**14**) also questions the interpretation applied to Guinier-Preston zones, claiming that the observed effects are really due to small nuclei of precipitate. Consequently, all of the property changes can be accounted for by various combinations of precipitate and accompanying strain, without reference to precipitation processes.

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[5] The existence of $\gamma'$ was first observed in 1934 by Wasserman (**11**) in considering the eutectoid decomposition of the $\beta$ phase in beryllium copper.

The present investigation attempts to correlate property changes occurring during aging with existing theories. At the same time, the influence of test direction is considered, since preliminary tests have demonstrated that aging may affect directionality. Although little is known regarding directional precipitation patterns, it is possible that such patterns could introduce some of the directionality observed. Geisler (9) and others have referred to precipitate in the form of platelets, so that the influence of precipitation might be more of a factor in one direction than another, or the rates of precipitation may vary in different directions.

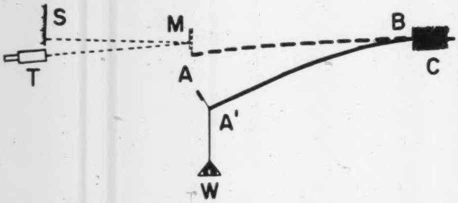

FIG. 1.—Bend Plasticity Arrangement for Measuring Permanent Set of Strip Specimen.

## MATERIALS

Three lots of ternary copper-beryllium-cobalt strip were employed. Compositions, thicknesses and cold-rolled reductions are listed in Table I. Small quantities of cobalt are added to the standard alloy to stabilize hardness response and to reduce grain boundary precipitate. The other elements represent unavoidable impurities present in commercial material.

Following penultimate cold reductions of 70 to 78 per cent, all lots were continuously (strand) solution treated at 1445 F. The ready-to-finish grain size averaged 0.030 mm.

## TEST METHODS

Four methods of test were employed: tension, electrical conductivity, diamond pyramid hardness and bending plasticity. Determinations of tensile strength, yield strength (0.2 per cent offset) and elongation (2-in. gage length) were in accordance with ASTM Methods of Tension Testing of Metallic Materials (E 8 – 54 T).[6] Proportional limit and E modulus values are difficult to obtain due to the curvilinear relationship between stress and strain that exists for beryllium copper (6,15). This condition becomes especially apparent when the tensile strength exceeds 100,000 psi.

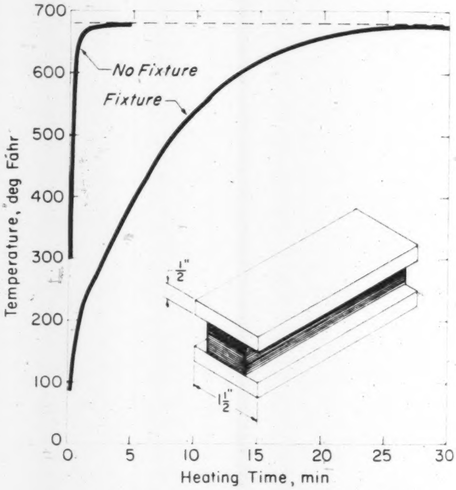

FIG. 2.—Relative Heating Rate of Beryllium Copper as Affected by a Fixture.

In order to provide an effective proportional limit that can be used for design and comparative purposes, a previously-described method (6) has been applied. A chord was drawn to the autographically recorded curve, so that there fell on the chord the initial points and last few values prior to the point where a definite yield or marked change of curvature could be noted. On the basis of prior evaluation in conjunction with more precise tests, this method was found to give a close approximation of the extent of the elastic region and agrees with elastic limits fixed by stress-set curves. Since the value of the proportional limit is dependent upon the sensitivity of the test method, it is qualified as the stress at 0.002 per cent offset from the chord. In this instance, 0.002 per cent offset represents the width of the autographic curve.

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[6] 1955 Book of ASTM Standards, Part 2, p. 1246.

The E modulus is taken as the tangent to the curve at zero stress. Although this value may not be completely realistic from a design standpoint, it offers a ready means for comparing the modulus of specimens having different proportional limits. Based upon past experience, the variation probably does not exceed ±1.5 per cent.

The bending plasticity method for determining permanent set is illustrated in Fig. 1 (16,17). Specimens 10 mm wide and about 190 mm long were sheared from the test strip in both longitudinal (0 deg) and transverse (90 deg) directions. These specimens were clamped horizontally at $B$ with clamp $C$, while weight $W$ was applied at $A$ for 1 min and then unloaded. One minute following unloading, the angle between the original and final positions of $AB$ was measured by telescope $A$, scale $S$, and galvanometer mirror $M$. The reported value for permanent set is the residual angle in radians resulting from a stress of 42,700 psi. This value is best obtained

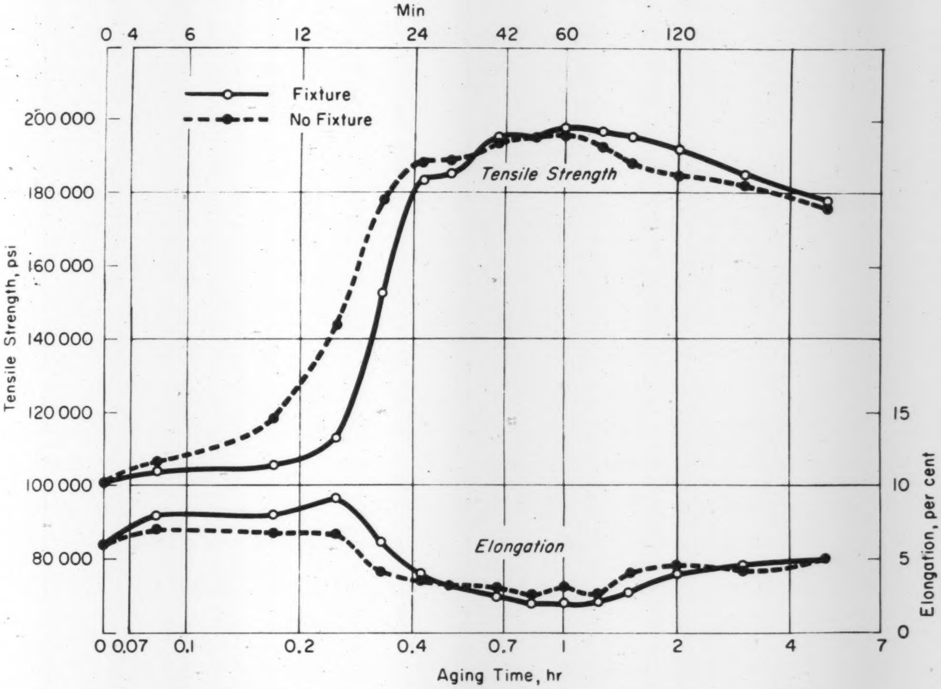

FIG. 3.—Influence of Fixture upon Aging Beryllium Copper Strip at 680 F.

by interpolation from a plot of maximum stress (at *B*) *versus* permanent set resulting from increasing the load from 0 to 150 g.

In order to insure the degree of flatness necessary in several of the tests, many of the specimens were age hardened between two cold-rolled steel plates. Since the mass effect of the fixture may be a factor, an attempt has been made to determine its influence upon heating rate.

The change in temperature with time was followed by means of thermocouples attached to loose specimens as well as imbedded in the center of 30 specimens stacked in the fixture. Figure 2 indicates relative heating rates from room temperature to 680 F. in a recirculating air furnace. Since cold specimens were charged into the furnace at temperature, the heating rate for loose specimens with no fixture is indicative of time required to bring the furnace back to temperature. In the case of fixture aging, position within the stack had no influence—based upon hardness and tension tests.

As might be expected, the quicker heating of loose specimens causes more rapid age hardening. Figure 3 shows that upon aging at 680 F, the fixture causes a lag of approximately 5 min, when considering tensile strength to peak hardness. The variation in elongation around 15 min is perhaps due to a temperature differential, causing substantial stress relief with little hardening for fixture specimens.

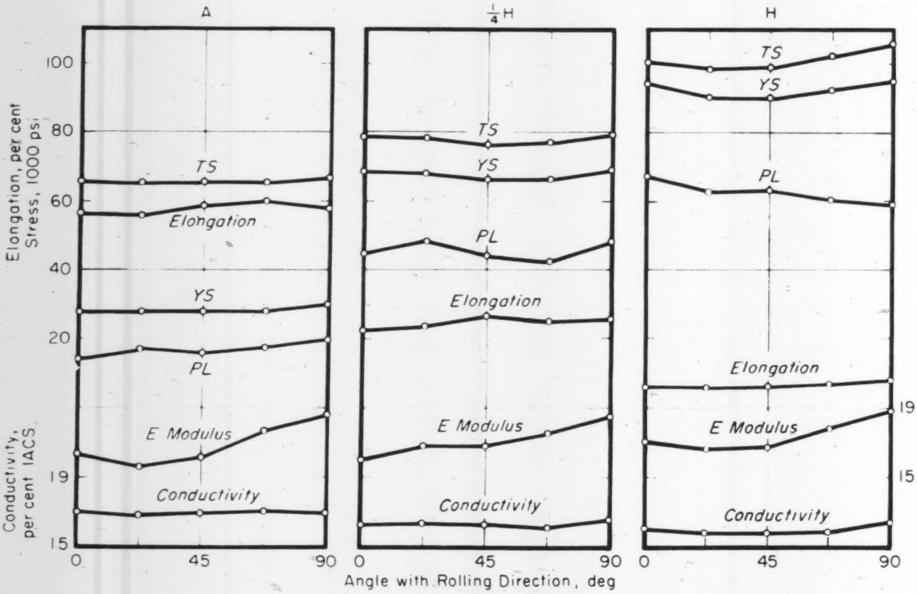

FIG. 4.—Effect of Grain Direction upon Properties of Heat-Treatable Beryllium Copper Strip.

## TEST RESULTS

The influence of temper rolling as well as age-hardening time and temperature upon directionality was studied. Although annealing temperature is known to have a marked effect upon preferred orientation in certain materials such as brass, the rather narrow solution-treating range for beryllium copper precludes wide variations in solution-treating temperature. Heavier cold reductions as generally associated with process rolling have not been evaluated in the present investigation. Values reported are averages resulting from two to six tests.

Light reductions, such as temper rolling, affect directionality. Figure 4 shows the influence of test direction upon solution-treated and cold-rolled strip (lot C), while Fig. 5 indicates the effect of age hardening for 3 hr at 600 F upon similar specimens.

The effect of aging temperature has been studied by heating specimens from lots A (solution treated) and B (half-hard) for 1 hr at temperatures ranging from ambient to 930 F. Specimens taken 0 and 90 deg with respect to the rolling direction were included. The results are shown in Figs. 6 and 7. A comparison of these two tempers, tested in the longitudinal direction only, is presented in Fig. 8.

To determine the influence of aging time, another series of tests were conducted on lot C strip in the solution-treated (A) and rolled hard (H) conditions. Specimens were aged in fixtures at 680 F for periods ranging from 5 min to 5 hr. Figure 9 shows the results for solution-treated strip, while similar data for rolled-hard material is given in Fig. 10. The influence of temper is indicated in Fig. 11 for tests conducted in the longitudinal (0 deg) direction.

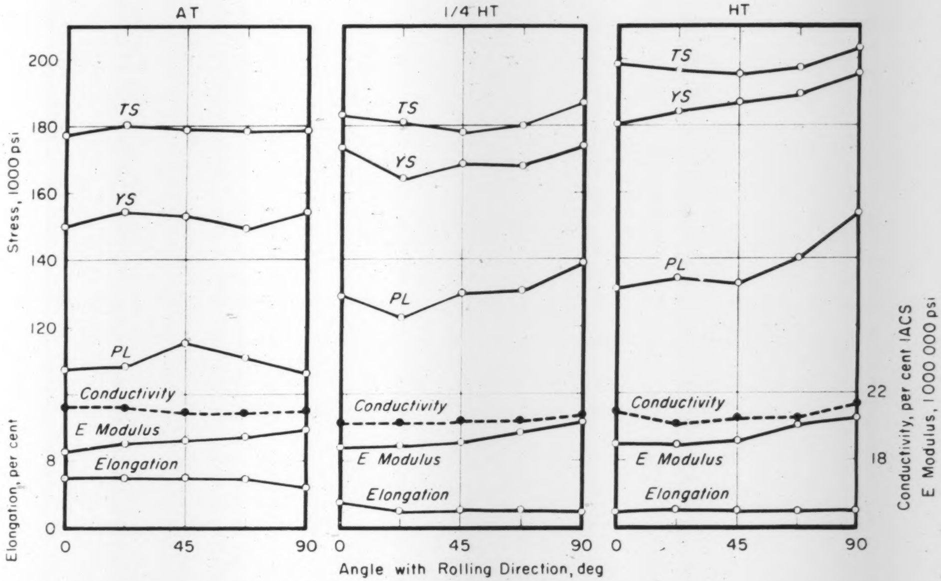

FIG. 5.—Effect of Grain Direction upon Properties of Beryllium Copper Strip Precipitation Hardened for 3 hr at 600 F.

## DISCUSSION OF DATA

The directional characteristics observed in beryllium copper strip result from cold work and heating. In the current investigation, cold work was limited to rolling in the form of temper reductions, while heating operations were confined to those temperatures associated with age hardening.

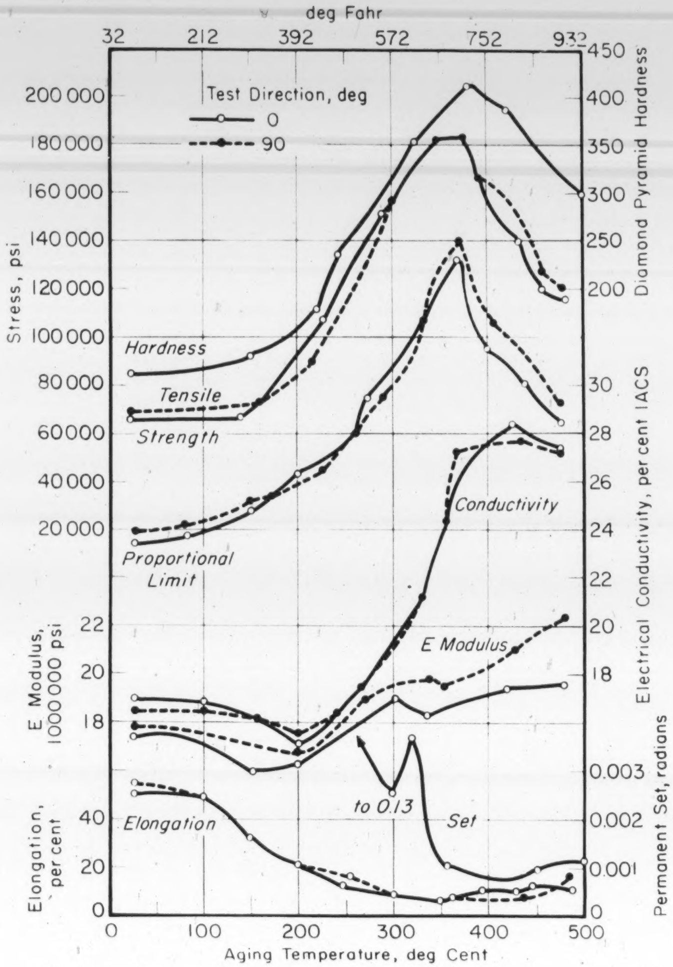

FIG. 6.—Effect of Aging Temperature (for 1 hr) upon Directional Properties of Solution Treated Beryllium Copper Strip.

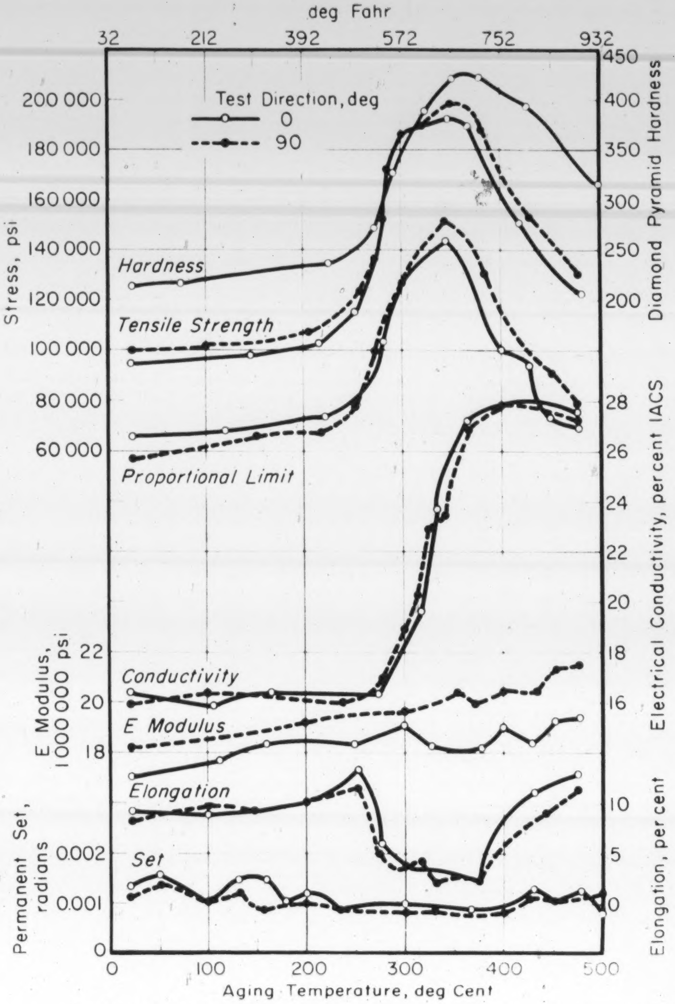

FIG. 7.—Effect of Aging Temperature (for 1 hr) upon Directional Properties of Half Hard Beryllium Copper Strip.

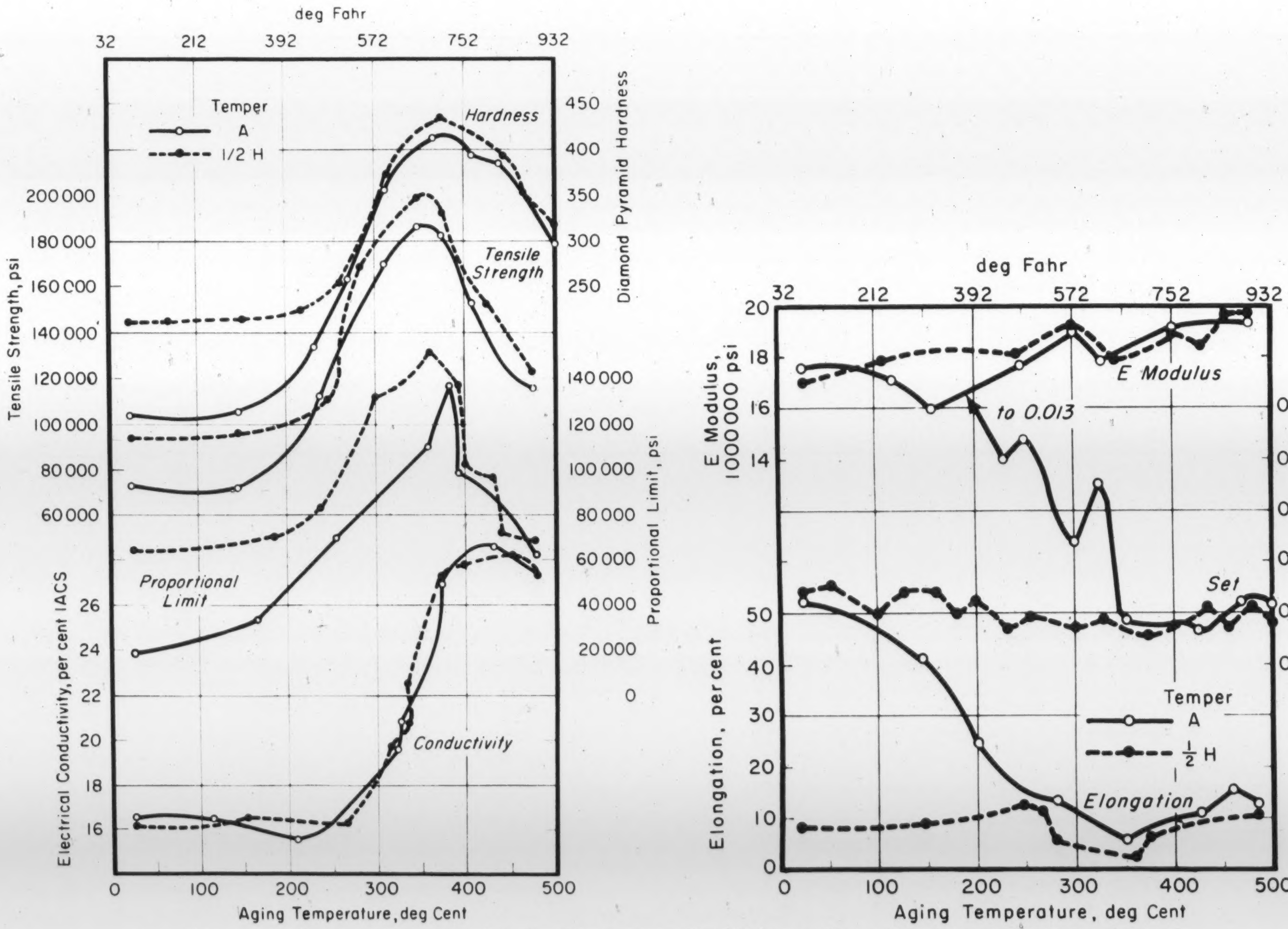

FIG. 8.—Influence of Temper and Aging Temperature (for 1 hr) upon Properties of Longitudina Specimens.

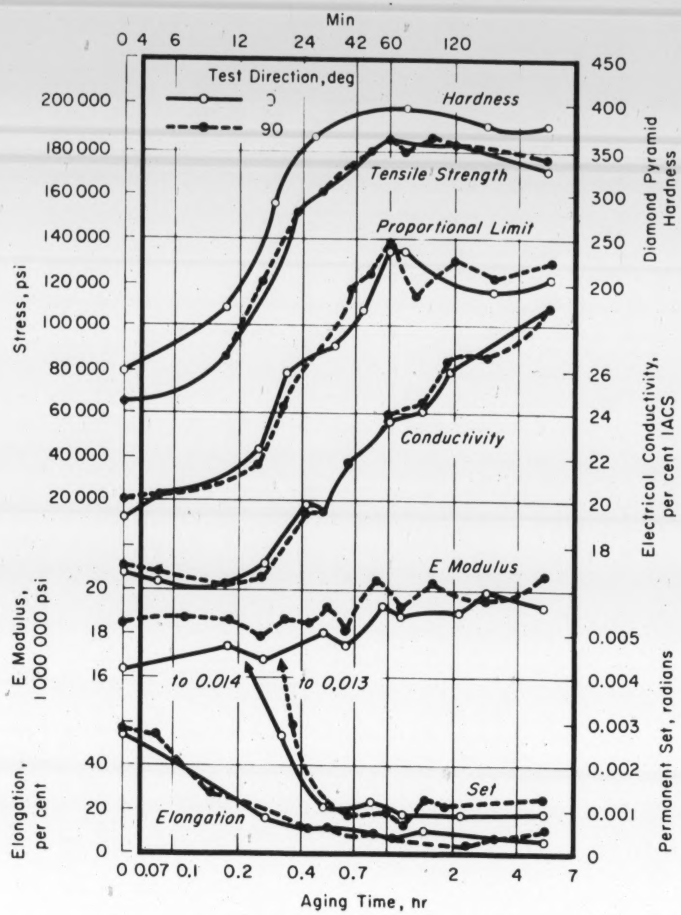

FIG. 9.—Effect of Aging Time at 680 F upon Directional Characteristics of Solution-Treated Beryllium Copper Strip.

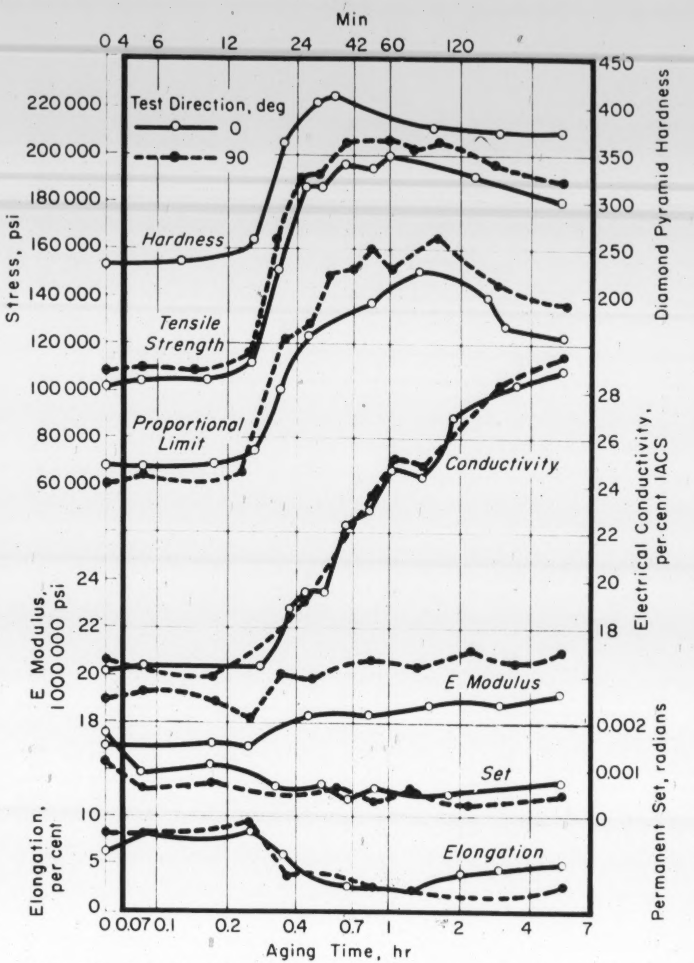

FIG. 10.—Effect of Aging Time at 680 F upon Directional Characteristics of Rolled-Hard Beryllium Copper Strip.

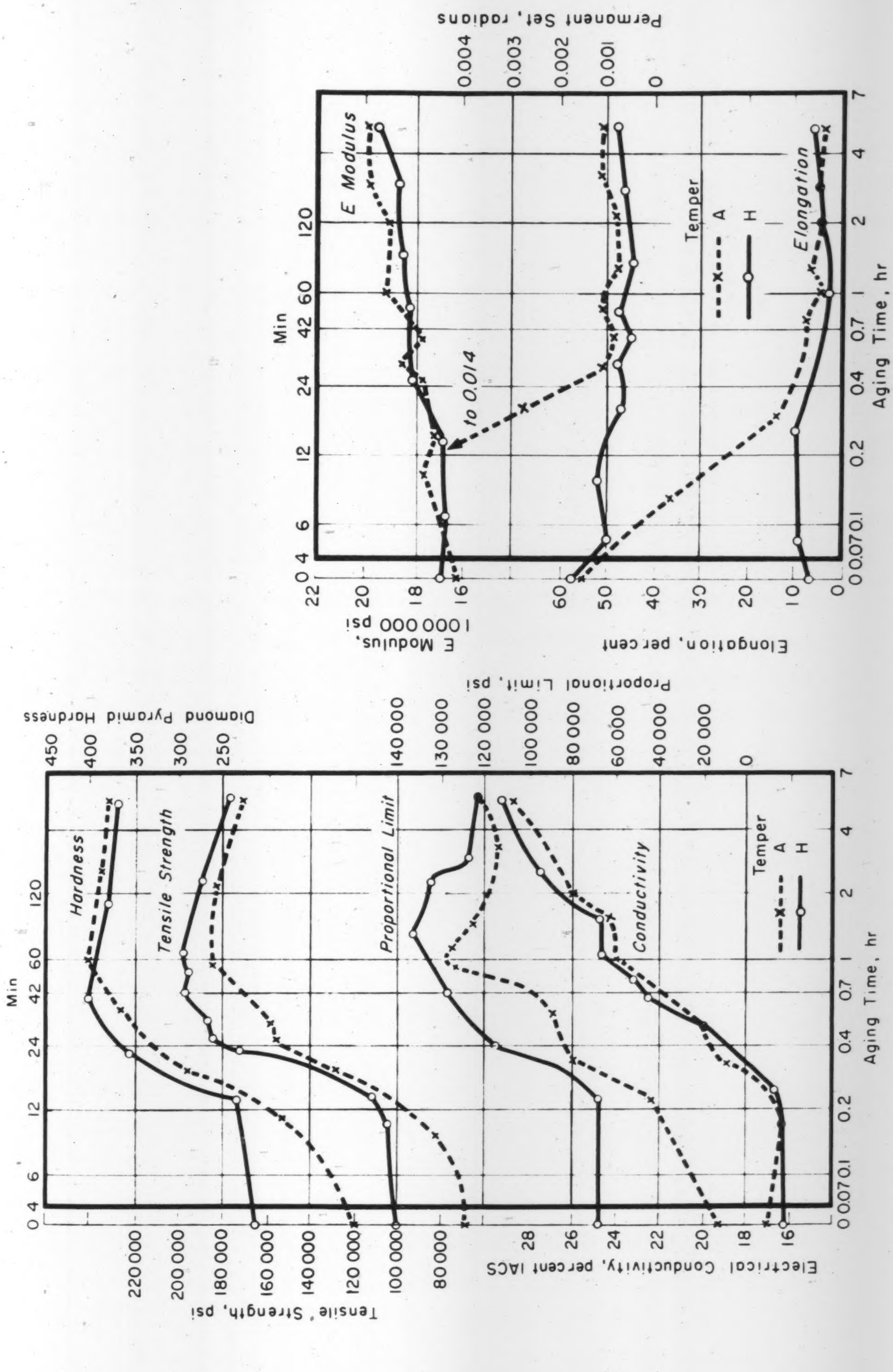

FIG. 11.—Influence of Temper and Aging Time at 680 F upon Properties of Longitudinal Specimens.

*Effect of Cold Rolling:*

In the case of heat-treatable strip (Fig. 4), the influence of test direction upon solution-treated (A) and quarter-hard ($\frac{1}{4}$ H) strip is negligible with the exception of E modulus. For hard-rolled (H) material, there is an appreciable increase in tensile strength and E modulus with the rolling direction. A steady drop in proportional limit can be noted with increasing angle, offering good agreement with earlier work on beryllium copper strip (4). With all tempers, conductivity is relatively uniform, although there is a definite drop with increasing cold work, as might be anticipated. For age-hardened strip (Fig. 5), an increase in angle of test produces increases in tensile and yield strength, proportional limit and E modulus although a dip or arrest frequently occurs at 45 deg. Conductivity and elongation exhibit no marked trend. Note the complete reversal of the proportional limit upon aging.

*Effect of Aging:*

Solution-treated beryllium copper strip shows little evidence of directionality. As indicated in Figs. 6 and 9, only a slight degree is present prior to aging. Although there are several reversals in directionality during aging, significant directional qualities are apparent only after aging at temperatures exceeding 570 F. E modulus provides an exception, since transverse (90-deg) values generally exceed those in the longitudinal direction.

Cold-rolled strip not only shows more directionality than solution-treated (A) stock but also presents a different pattern, as indicated in Figs. 7 and 10. Before aging, transverse tensile strength and E modulus are substantially higher than longitudinal, while the reverse is true for proportional limit. In the case of permanent set, test direction does not appear to be an important factor, although it is of interest to note that there is usually slightly less set in the transverse direction.

With increasing aging times or temperatures, directionality as measured by tensile strength and proportional limit steadily decreases until it is negligible within the range of most rapid hardening. Upon reaching peak properties, directionality becomes apparent again and generally increases upon further aging.

Several other features of Fig. 7 merit consideration. For example, there is a sudden change in directionality as indicated by electrical conductivity accompanying peak strength at 570 F. In addition, transverse E modulus is substantially higher than its longitudinal counterpart at all aging temperatures, although variations occur which correspond with certain changes in directionality noted for some of the other properties. No directional effects are observable in the case of elongation except at higher temperatures, agreeing with the marked directionality found in tensile properties beyond maximum strength.

*General:*

A comparison of the effects of cold work and heat treatment is afforded by Figs. 8 and 11. Of particular interest is the rapid aging response of solution-treated stock as compared with rolled tempers. It is generally believed that cold work creates points within the crystal lattice of lowered activation energy for the creation of nuclei, thereby resulting in a more rapid precipitation rate. In the present investigation, a faster initial response is observed in the case of solution-treated strip. The aging response of cold-worked strip, after an initial period of retardation, proceeds at an extremely rapid rate, eventually sur-passing that of the solution-treated material. Consequently, the peak for half-hard material occurs at a slightly lower time or temperature than the peak for solution treated strip. On account of overaging, curves for rolled material fall rapidly to the level of the solution-treated condition, so that above 705 F there is no real difference in hardness or tensile properties for the two conditions.

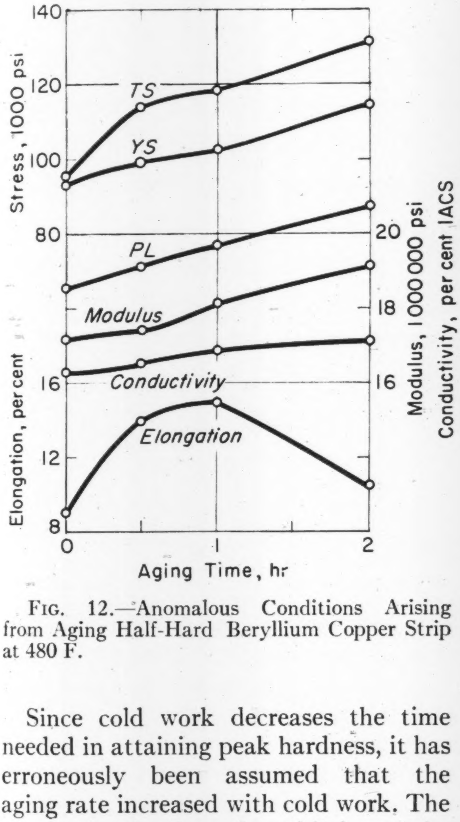

FIG. 12.—Anomalous Conditions Arising from Aging Half-Hard Beryllium Copper Strip at 480 F.

Since cold work decreases the time needed in attaining peak hardness, it has erroneously been assumed that the aging rate increased with cold work. The curves clearly show that this is not the case, because initial hardening occurs much more rapidly in solution-treated material. Similarly, Gruhl and Wassermann (**8**) have observed that cold deformation may in some way inhibit precipitation. In fact different hardening processes may occur, since Geisler (**9**) found no evidence of transition phases for cold-deformed material, while Gruhl and Wassermann noted a similar absence of the transition phase as well as suppression of Guinier-Preston zones.

Elongation follows the usual pattern for an age-hardening alloy, except for the rise at 480 F in the case of half-hard strip. At first glance this effect, which is also shown clearly in Fig. 8(*b*), seems due to stress relief, since the conductivity curve suggests that precipitation is negligible or perhaps has not occurred below 480 F. The slight increases in hardness, strength, conductivity, and modulus that take place below 390 F are similar in magnitude to those noted in brass, bronze, and copper for approximately the same temperature range. The more rapid rise in hardness and strength coupled with a slight drop in conductivity and E modulus between 390 F and 480 F suggests hardening caused by matrix strain. Because greater matrix strain would be expected to decrease elongation, additional work is needed to clarify this anomalous condition.

As a means of further checking this situation, supplementary tests were conducted. Duplicate specimens were heated for 0, $\frac{1}{2}$, 1, and 2 hr at 480 F. Figure 12 indicates an even more pronounced effect, since aging for 1 hr produced an increase in elongation from 9 to 15 per cent instead of the 9 to 12.5 per cent rise obtained in the earlier tests. Aging for 2 hr causes a sharp drop in elongation combined with further increases in strength, modulus and conductivity.

In this instance, the individual roles played by stress relief, matrix strain, and actual precipitation are difficult to evaluate. It is unlikely that these changes result from precipitation. A partial explanation may be found in possible orientation changes mentioned above (**2,3**). In addition, stress relief or recovery from lattice distortion may occur in conjunction with orientation to produce these changes.

As previously indicated, the permanent set reported results from a bending stress of 42,700 psi. Since the effective proportional limit has been shown to approximate the elastic limit for beryllium copper (6), it is to be expected that specimens having a proportional limit greater than 42,700 psi would exhibit negligible permanent set. The proportional limit for the solution-treated condition exceeds this value above 390 F (Fig. 6), but appreciable set remains up to 660 F—the point of maximum tensile strength. Although small amounts of permanent set are difficult to detect in tension, measurable set has been observed in deflection tests at extremely low levels of stress (18).

Two anomalous trends are apparent in the case of electrical conductivity: (1) the drop of the solution-treated curve at 390 F and (2) the decrease in conductivity above 795 F, as shown in Fig. 8(*a*). It is also of interest to note that conductivity for solution-treated material starts to increase more rapidly and at a lower temperature than it does for half-hard strip, but at 615 F this trend is reversed. Furthermore, the improvement in conductivity occurs at a higher temperature than the initial increase in hardness and tensile properties.

A number of investigators (8,13,14,19) have observed the unexpected decrease in electrical conductivity resulting from aging at low temperatures or short periods at higher temperatures. This decrease is generally attributed to strain prior to actual precipitation. As shown in Figs. 8 and 11, the decrease in conductivity is of the same magnitude as produced by cold rolling, but this change is small compared with that caused by depletion of the solid solution. Assuming that conductivity is sensitive to changes of composition, Figs. 8 and 11 indicate similar precipitation rates for all tempers. This suggests that the initial hardening of solution-treated strip may not be due to precipitation.

On first glance, the leveling off and eventual drop in conductivity as shown in Figs. 6, 7, and 8 at temperatures above 750 F seems surprising. Although there is no drop in modulus at this temperature, the gradual rise noted at lower temperatures appears to be arrested. Other investigators (8,20) have observed this same trend.

Perhaps the most likely cause of this phenomenon is to be found in the increase of solubility with rising temperatures. For example, Gruhl (21) has measured a solubility limit of 0.19 per cent beryllium at 570 F but 0.98 per cent at 930 F. This means that less $\gamma$ can precipitate at higher temperature, so that the hardening potential is decreased. Masing and Haase (20) have demonstrated the validity of this assumption, since both conductivity and modulus initially aged at 1020 F improved upon subsequent heating at 660 F. Resolution of $\gamma$ may also be accompanied at the higher aging temperatures by recrystallization of the matrix, producing a change in directionality through reorientation.

E modulus is affected by the same factors (internal stress, preferred orientation and composition) that influence electrical conductivity, but not necessarily to the same degree. For example, preferred orientation causes much greater variation in elastic modulus than conductivity, while the reverse seems to be true for composition. Internal stress, on the other hand, has a slight effect upon both properties.

In the present investigation, all three of these factors are operative during the aging of beryllium copper. The role of preferred orientation, for instance, is known to be an important one; however, the influence of the various textures resulting from cold rolling and solution treating has not been established, although the presence of directionality is indicated by other test properties. Furthermore, preferred orientation and internal stress both result from cold rolling, and both may be altered or removed by heating, so that evaluation of these effects individually is difficult.

The influence of composition is perhaps better understood. By raising the solute composition of a solid solution (such as beryllium content in the $\alpha$ solid solution) the E modulus is decreased (22). With precipitation of a second phase (in this case $\gamma$), the effective E modulus depends upon the moduli of the two phases and the degree to which each is present (23). Consequently, when precipitation occurs, the decrease of beryllium in solution will cause a slight improvement in modulus, while the precipitation of high-modulus $\gamma$ should produce a further increase. Guillet (24,25) has reported an E modulus of 25,600,000 psi for $\gamma$ compared to 17,100,000 psi for the $\alpha$ phase, so that the improvement due to precipitation of $\gamma$ far exceeds that caused by a decrease in solute content. Consequently, the over-all increase in E modulus observed during age hardening probably results from the presence of $\gamma$ precipitate, while the local fluctuations may be due to various combinations of internal stress and orientation.

It is of interest to note that the hardening effect due to aging decreases with increasing temper and, for a given cold reduction, decreases with increasing aging temperature. In the case of higher aging temperatures, several factors are present that could lead to reduced hardening. Gruhl and Wassermann (8) have suggested a different aging mechanism, since the transition phase was not found upon aging at temperatures above 570 F. In addition, the increase in solubility with increasing temperatures causes a potential decrease in hardening effect, due to the reduction in the quantity of beryllium available for precipitation in the form of $\gamma$. Retrogression may also be a factor (26), because its influence increases at higher temperatures. Since the degree of stress relief improves with increasing temperatures, it is probable that stress relief reduces the relative hardening effect in cold-worked materials.

Data presented indicate that precipitation hardening does not necessarily proceed at the same rate nor at the same time for different angles with respect to the rolling direction. For cold-rolled material, the improvement in tensile properties is greater in the transverse than the longitudinal direction. All tempers show greater stiffness (higher modulus) across the strip. Unfortunately, it is not possible to judge the exact role of actual precipitation, since recovery from lattice distortion and possible alterations in orientation may be responsible in part for the changes in directional characteristics noted during heating in and below the usual age-hardening range for beryllium copper.

Of the various properties evaluated, modulus provides perhaps the most sensitive index. Changes in directionality which produce small variations in other properties may cause marked changes in modulus. Careful determinations of modulus provide data of sufficient precision to evaluate directionality.

## CONCLUSIONS

1. The directional characteristics of beryllium copper are affected by cold rolling, solution treating, and precipitation hardening. The role of precipitation, although important, has apparently received little attention in the past.

2. Factors contributing to the directionality noted include lattice distortion,

recrystallization, and precipitation patterns. Since overlapping generally occurs, separate evaluation of these factors is difficult.

3. Although certain transverse properties may be inferior in the heat-treatable condition, after aging the transverse characteristics are equal to or better than the longitudinal properties. Tensile properties including proportional limit and yield strength are generally superior in the transverse direction, while modulus may be as much as 15 per cent greater across the strip. Resistance to permanent set resulting from deflection is generally superior in the transverse direction.

4. Of the properties studied, modulus provides perhaps the most sensitive index of directionality, while electrical conductivity is the least affected.

5. Contrary to general belief, more rapid initial hardening was observed for solution-treated material as compared with cold-rolled strip during aging. Following a period of initial retardation, the aging rate for cold-worked material surpasses that of solution-treated strip, so that maximum hardness occurs at slightly lower temperatures or shorter aging times than solution-treated strip.

6. The initial phases of hardening, which are generally accompanied by a decrease in modulus and conductivity in the case of solution-treated material, may not result from actual precipitation but rather a complex combination of lattice strain, stress relief and preferred orientation. The role of transition phases cannot be evaluated, since property data for these phases is lacking.

## REFERENCES

(1) M. Cook, T. L. Richards, and G. F. Bidmead, "Influence of Cold Deformation on the Young's Modulus of Some Non-Ferrous Metals," *Journal*, Inst. Metals, Vol. 83, pp. 41–47 (1954–1955).

(2) G. S. Farnham and H. O'Neill, "Crystal Re-Orientation on Heating Drawn Copper Wires," *Journal*, Inst. Metals, Vol. 55, pp. 201–208 (1934).

(3) M. Kawasaki, "The Effect of the Low-Temperature Annealing upon the Fibrous Structures of Highly Cold-Worked Copper," *Science Reports of the Research Institutes*, Tohoku Univ., Vol. 2, pp. 861–870 (1950).

(4) J. T. Richards and E. M. Smith, "The Forming Characteristics of Beryllium Copper Strip," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 1085–1100 (1950).

(5) Ivan Cerkesov, "Spring Grade Beryllium Alpha Bronze," *Hutnicke Listy*, Nos. 3 and 4, pp. 115–120, pp. 186–190 (1952).

(6) J. T. Richards, R. K. Levan, and E. M. Smith, "The Influence of Cold Work and Heat Treatment on the Engineering Properties of Beryllium Copper Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, pp. 771–789 (1951).

(7) J. T. Richards, "The Influence of Preferred Orientation upon the Properties of Beryllium Copper Wire," in preparation.

8) W. Gruhl and G. Wassermann, "The Course of Precipitation in Copper-Beryllium Alloys," *Metall*, Vol. 5, pp. 93–98, pp. 141–145 (1951).

(9) A. H. Geisler, J. H. Mallery, and F. E. Steigert, "On the Mechanism of Precipitation in Copper-Beryllium Alloys," *Journal of Metals*, Vol. 5, pp. 307–316 (1952).

(10) T. Morinaga, T. Takahashi, and T. Takenaka, unpublished work presented at Japan Inst. of Metals, Oct. 1951 and April 1952.

(11) G. Wassermann, "Transformations in Eutectoid Alloys, "*Zeitschrift fur Metallkunde*, Vol. 26, pp. 256–259 (1934).

(12) A. Guinier and P. Jacquet, "Study of the Hardening of Copper-Beryllium Alloys," *Revue de Metallurgie*, Vol. 41, pp. 1–16 (1944).

(13) A. G. Guy, C. S. Barrett, and R. F. Mehl, "Mechanism of Precipitation in Alloys of Beryllium in Copper," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 175, pp. 216–238 (1948).

(14) A. H. Geisler, "Precipitation from Solid Solutions of Metals," "Phase Transformations in Solids," pp. 387–544, John Wiley and Sons, New York, N. Y. (1951).

(15) J. T. Richards, "An Evaluation of Several Static and Dynamic Methods for Determining Elastic Moduli," Symposium on Determination of Elastic Constants, Am.

Soc. Testing Mats., pp. 71–100 (1952). (Issued as separate publication *ASTM STP No. 129*).

(16) K. Murakawa and N. Takashima, "Bending Plasticity of Beryllium-Copper Alloy Sheets," *Bulletin*, Aeronautical Research Inst. (Japan), No. 244, pp. 437–442 (1944).

(17) K. Murakawa, "Bending Elasticity of Slice Sheets of Metals and Hardening by Precipitation of Copper Alloys," *Reports of the Institute of Science and Technology*, University of Tokyo, pp. 6–8, 17–20, 60–62, 177–180 (1947).

(18) H. G. Williams, "Test Methods for Predicting Diaphragm Performance," *Instrument Maker*, Vol. 14, March–April, 1946.

(19) G. Masing and O. Dahl, "Changes in the Electrical Conductivity and Volume During the Age-Hardening of Beryllium-Copper Alloys," *Beryllium*, Siemens-Konzern, Berlin (Germany), translated by R. Rimbach and A. J. Michel, Chemical Catalog Co., New York, N. Y., pp. 151–170 (1932).

(20) G. Masing and C. Haase, "The Changes in the Modulus of Elasticity During the Age-Hardening of Beryllium-Copper Alloys," *Beryllium*, Siemens-Konzern, Berlin (Germany) translated by R. Rimbach and A. J. Michel, Chemical Catalog Co., New York, N. Y., pp. 171–179 (1932).

(21) W. Gruhl, "The Course of Precipitation of Copper-Beryllium at High Temperatures," *Metall*, Vol. 7, pp. 978–982 (1953).

(22) W. Koster and W. Rauscher, "Relations Between the Modulus of Elasticity of Binary Alloys and Their Structure," *Zeitschrift fur Metallkunde*, Vol. 39, pp. 111–120 (1948).

(23) B. F. Middleton, "Distribution of Stress in the Vicinity of a Spherical Inclusion," *Technical Note No. M. 9546*, Royal Aircraft Establishment (1945).

(24) L. Guillet, "On the Modulus of Elasticity of Copper-Beryllium Alloys," *Comptes Rendus*, Vol. 201, pp. 960–962 (1935).

(25) L. Guillet, "Contribution to the Study of the Elastic Modulus of Metallic Alloys," *Revue de Metallurgie*, Vol. 36, pp. 497–521 (1939).

(26) W. Gruhl and U. Gruhl, "Retrogression in Copper-Beryllium Induced by Cold Deformation," *Metall*, Vol. 8, pp. 20–23 (1954).

# EFFECT OF CERIUM ON THE HEAT TREATMENT RESPONSE OF PRECIPITATION-HARDENING TITANIUM ALLOYS

By Ralph H. Hiltz, Jr.[1] and Nicholas J. Grant[1]

## Synopsis

A study was made of the effects of combining the weak precipitation-hardening alloys of titanium, titanium-silver, titanium-copper, and titanium-silicon with the more intense precipitation hardening but hot short titanium-cerium alloys. Aging temperatures were from 200 to 600 C for times of ½ to 4 hr. Significant aging response was noted in the ternary alloys titanium-silver-cerium, titanium-copper-cerium, and titanium-silicon-cerium, the latter alloy showing a double hardness peak. The alloy of titanium with copper and cerium shows pronounced hardening and slow overaging.

The development of alloys of titanium has progressed at a very rapid rate since the metal's inception as a structural material. The development of sheet alloys, however, especially those suitable for elevated temperature applications, has lagged behind the development of other types of alloys. Several directions of study are available for developing such alloys. Age hardening alpha-beta alloys and solid solution alloys have received a great deal of attention to date, leaving precipitation-hardening or dispersion-hardening alloys as the most logical direction at this time.

Four binary titanium systems offer potential precipitation-hardening alloys; titanium-copper, titanium-silver, titanium-silicon and titanium-cerium.

Precipitation hardening of titanium-copper alloys has been studied by Holden, Watts, Ogden, and Jaffee (**1**)[2] and Howe, Saarema, and Parr (**2**). The results of these investigations have shown that the alloys are amenable to precipitation hardening, but that peak hardness occurs in less than 10 min at temperature. The rapid aging effect appears due to the nonsluggish nature of the compound formation and the shape of the alpha solvus, as in Fig. 1, which shows very little recession from maximum solubility down to room temperature.

Titanium-silver alloys have not undergone any formal study of precipitation-hardening characteristics. This lack of investigation is due to the uncertainty of the phase diagram. Three diagrams exist (**3, 4, 5**) which differ significantly in the limit of alpha solid solubility. The solubility difference among the phase diagrams at 800 C is from about 2 to 12 per cent silver by weight. The diagram of Adenstedt and Freeman (4) is shown in Fig. 2. There has been a general belief that titanium-silver alloys would give a precipitation-hardening response similar to titanium-copper alloys.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 820.

Titanium-silicon alloys have been investigated by Antes and Edelman (6). Their results show a very shallow hardening of these alloys with precipitation-hardening treatments. The titanium-silicon system (6) (Fig. 3) also shows a shallow alpha solvus slope. The slow diffusion rate of silicon and the slow rate of formation of the compound $Ti_5Si_3$ cause the increase in hardness to be spread over a long time interval rather than over a short interval, as in the copper system.

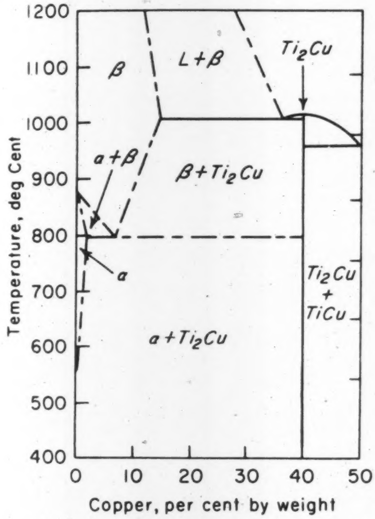

FIG. 1.—Precipitation Hardening Characteristics of Titanium-Copper Alloys (**10**).

In all of the above systems, precipitation hardening occurs, but is not of sufficient magnitude or controllability to be practical. Titanium-cerium alloys, however, studied extensively by Taylor (7,8) (Fig. 4), do exhibit appreciable precipitation-hardening response. These alloys exhibited severe hot shortness which offset their advantageous heat-treatment response. Since cerium forms no compounds with titanium, the hot shortness was blamed on the presence of elemental cerium at grain boundaries. Studies at Watertown Arsenal Laboratories (9) of embrittled titanium alloys containing cerium revealed that in alloys containing aluminum, elemental cerium was not the responsible agent.

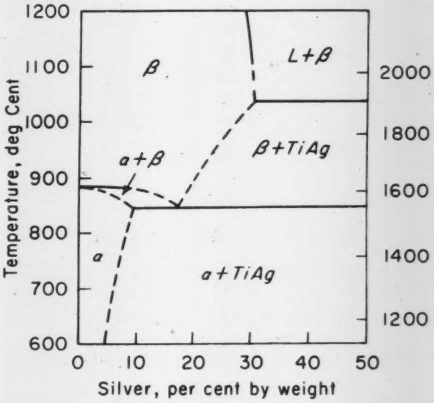

FIG. 2.—Precipitation Hardening Characteristics of Titanium-Silver Alloys (**4**).

The hot shortness was due to the presence of a hard intermetallic phase, tentatively identified as a face-centered cubic compound of the composition $Al_4Ce$. This phase, probably formed in the melt, showed great reluctance to dissolve at any temperature below the alpha transus. The phase could be dissolved in the beta phase; and titanium-cerium alloys could be forged if the forging temperature was above 2400 F. The inability to dissolve the compound in the alpha phase prevented any exploitation of potential precipitation-hardening characteristics. The appearance of the phase did, however, indicate that the precipitation-hardening potential of titanium-cerium alloys might be realized by adding a second solute element. Under the right conditions, a compound of cerium and the second solute element would be precipitated rather than elemental cerium. The elements of interest to add as the second solute are those also showing some precipitation hardening in combination with titanium, namely, silver, copper, or silicon. A preliminary investigation of the precipitation-hardening response of a titanium-cerium alloy with additions of copper, silicon, or silver by means of microhardness testing was thus undertaken.

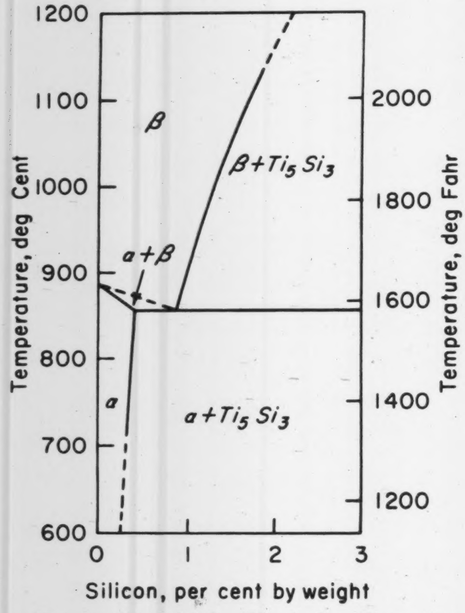

FIG. 3.—Precipitation Hardening Characteristics of Titanium-Silicon Alloys (**11**).

### MATERIALS

*Base Materials:*

The alloys were prepared from Kroll Process titanium sponge (Brinell hardness number, 110) and high purity (99.9+) alloy additions, including the cerium.

*Preparation of Alloys:*

All alloys were prepared as 100-g charges in lots of five, to provide a final amount of 1 lb for each alloy. The weighing of all materials was done on an analytical balance to insure uniformity of alloy content between each 100-g charge. Cerium alloying additions were cut slightly in excess of the desired weight and polished to size on fine silicon carbide papers just prior to melting, to minimize the addition of cerium as cerium oxide. The percentages by weight of silver, silicon, and copper added to the various alloys were just short of maximum solid solubility, using the titanium-silver phase diagram according to Adenstadt and Freeman (4) as the most reliable. Cerium, 0.5 per cent

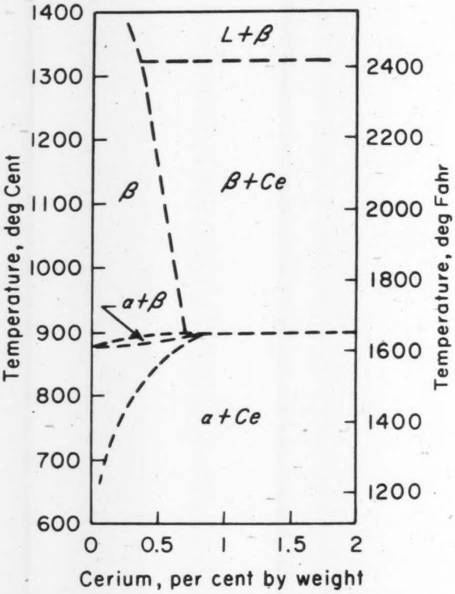

FIG. 4.—Precipitation Hardening Characteristics of Titanium-Cerium Alloys (**8**).

by weight, was added. Since the titanium-cerium system shows a peritectoid reaction, the maximum amount of cerium could not be used. A solution temperature corresponding to maximum solubility would place the alloy in the alpha + beta field if 100 per cent recovery of the cerium during melting was not realized. If 0.5 per cent cerium is added, the solution temperature corresponds to the transformation temperature of pure titanium, and the alloy will be in the alpha field no matter what the recovery of cerium.

TABLE I.—NOMINAL AND ACTUAL CHEMICAL COMPOSITION OF ALLOYS.

| | Cerium per cent | Copper, per cent | Silicon, per cent | Silver, per cent | Oxygen, per cent | Nitrogen, per cent | Iron, per cent | Carbon, per cent | Hydrogen, per cent |
|---|---|---|---|---|---|---|---|---|---|
| Titanium-Cerium | Nominal 0.5 | ... | ... | ... | ... | ... | ... | ... | ... |
| | Actual 0.42 | ... | ... | ... | ... | ... | ... | ... | ... |
| Titanium-Copper | Nominal | 2.00 | ... | ... | ... | ... | ... | ... | ... |
| | Actual | 2.04 | ... | ... | ... | ... | ... | ... | ... |
| Titanium-Silicon | Nominal | ... | 0.50 | ... | ... | ... | ... | ... | ... |
| | Actual | ... | 0.45 | ... | ... | ... | ... | ... | ... |
| Titanium-Silver | Nominal | ... | ... | 7.00 | ... | ... | ... | ... | ... |
| | Actual | ... | ... | 6.50 | ... | ... | ... | ... | ... |
| Titanium-Copper-Cerium | Nominal 0.5 | 2.00 | ... | ... | ... | ... | ... | ... | ... |
| | Actual 0.41 | 2.04 | ... | ... | ... | ... | ... | ... | ... |
| Titanium-Silver-Cerium | Nominal 0.5 | ... | ... | 7.00 | ... | ... | ... | ... | ... |
| | Actual 0.42 | ... | ... | 6.48 | ... | ... | ... | ... | ... |
| Titanium-Silicon-Cerium | Nominal 0.5 | ... | 0.50 | ... | ... | ... | ... | ... | ... |
| | Actual 0.40 | ... | 0.48 | ... | ... | ... | ... | ... | ... |
| Titanium | ... | ... | ... | ... | 0.127 | 0.009 | 0.18 | 0.07 | 0.0015 |

All alloys were melted in a nonconsumable tungsten-arc furnace under a positive pressure of purified argon. In all, eight compositions were prepared: the three ternary systems, the four pertinent binary systems, and pure titanium. The pure titanium and the binary alloys were prepared as reference material. The nominal and actual compositions are given in Table I. The impurity content is listed only for the pure titanium but is representative of all melts except those containing cerium, where the oxygen level is slightly higher due to the presence of $CeO_2$. Deviation between buttons of each alloy was a maximum of ±0.02 per cent even in the case of cerium.

## Test Procedure

The buttons were cold-worked to 50 per cent reduction by press forging and then cut into $\frac{1}{4}$-in. slices. The end pieces which received less than 50 per cent reduction due to the roundness of the buttons were used for chemical analysis. Slices from each alloy group were heated in salt at 850 C for various time periods. The minimum time for recrystallization and solution of the second phase was found to be 1 hr at this temperature. A single-phase structure was never attained in the alloys containing cerium due to the presence of $CeO_2$.

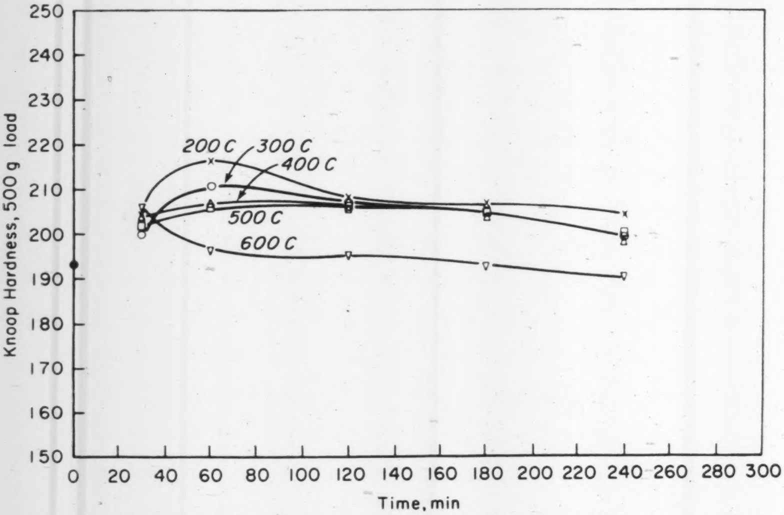

FIG. 5.—Precipitation Hardening Response of Commercially Pure Titanium at Various Temperatures.

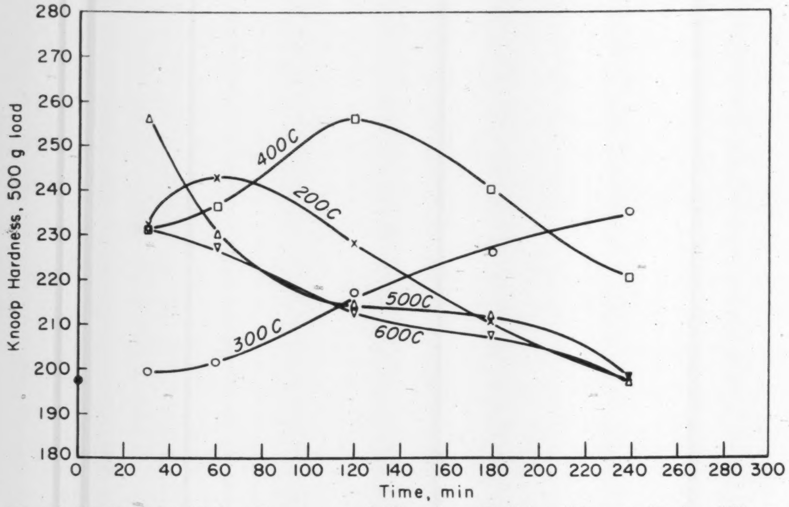

FIG. 6.—Precipitation Hardening Response of Titanium-Cerium Alloy at Various Temperatures.

For the aging studies, all slices from each alloy were heated in salt for 1 hr at 850 C and water-quenched. Specimens were divided into groups for aging. Aging was carried out in salt at temperatures ranging from 200 to 600 C and for times from $\frac{1}{2}$ to 4 hr. Each aging treatment was performed in duplicate. Specimens were air cooled from the aging temperature. Each specimen was examined microscopically on a cut face. The hardness of each specimen was measured, employing a Wilson microhardness tester, a Knoop indentor and a 500-g load. Ten readings were taken on each specimen, consecutively along the longitudinal axis. Readings were statistically analyzed in terms of 95 per cent probability, and all readings falling outside these limits were rejected. The remaining readings were averaged and this value taken as the hardness of each specimen. Deviation between duplicate specimens was a maximum of ±2 Knoop hardness numbers. Hardness values were plotted against aging time to show the precipitation-hardening response.

One specimen from each alloy system was sealed in Vycor tubing under vacuum, solution treated, and aged at 500 C for 170 hr to agglomerate the precipitating phase. This was done to allow observation of the distribution and shape of the particles and to reduce particle size X-ray line broadening in subsequent X-ray analysis. These specimens were examined microscopically. Following a metallographic study, small pieces were cut from each specimen and brominated at 200 C. In an atmosphere of bromine vapor, titanium is converted to $TiBr_4$, which as a gas is swept from the system by passage of helium. Stable compounds are not affected by the bromine and remain behind. Cerium if present as the element is converted to $CeBr_3$, which also remains as part of the residue. X-ray diffraction patterns were obtained from the powder residues using a 57.3-mm Debye-Scherrer camera and $CuK_2$ radiation at 50 kv and 20 ma.

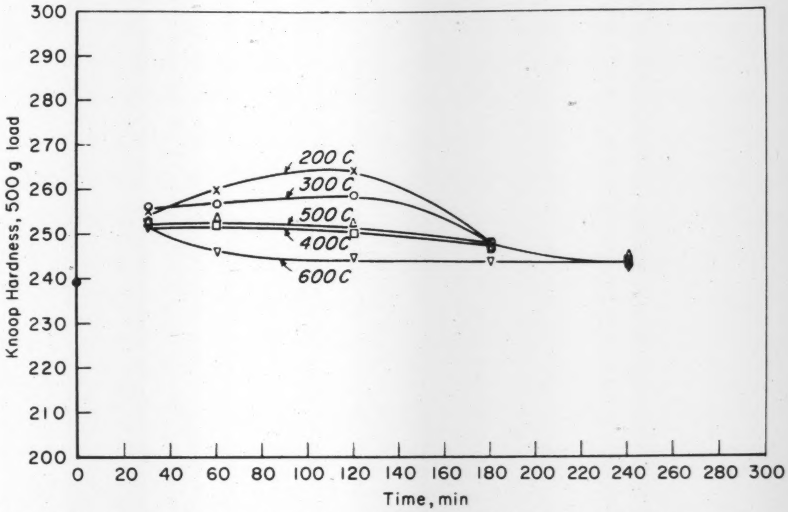

FIG. 7.—Precipitation Hardening Response of Titanium-Copper Alloy at Various Temperatures.

## TEST RESULTS

### *Hardness Results:*

The pure titanium material shows a rather small precipitation-hardening response at low aging temperatures. This increase is probably due to the precipitation of carbides and hydrides. At higher aging temperatures where diffusion is faster and the solubility of carbon

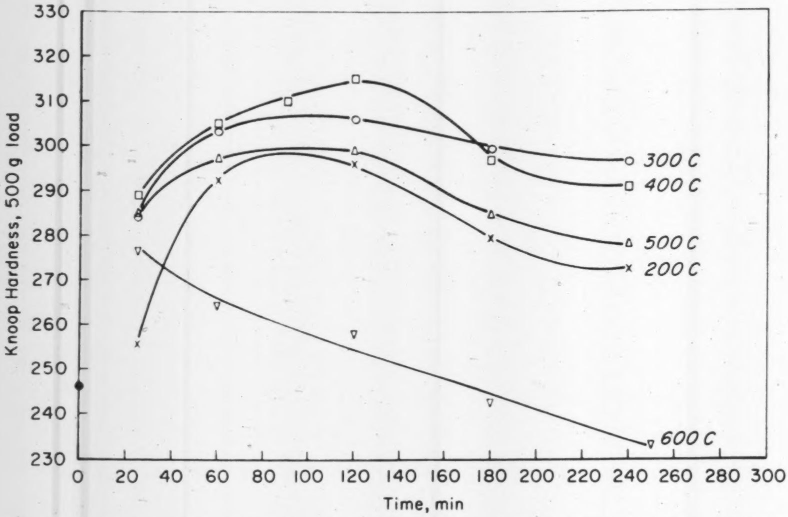

FIG. 8.—Precipitation Hardening Response of Titanium-Copper-Cerium Alloy at Various Temperatures.

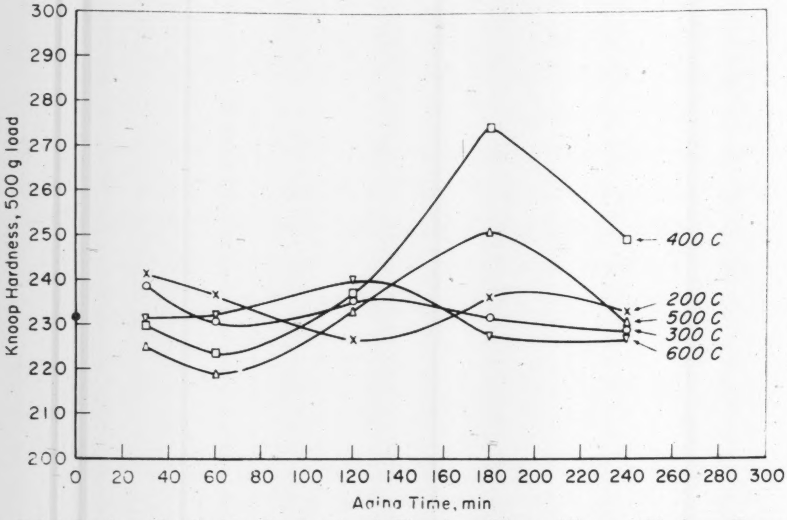

FIG. 9.—Precipitation Hardening Response of Titanium-Silver Alloy at Various Temperatures.

larger, the response decreases. These results are shown in Fig. 5.

The response of the titanium-cerium binary alloy is much the same as that reported by Taylor (7) except for the data at 200 C. The anomaly in the data, as shown in Fig. 6, is partially explainable in terms of hardening by precipitation of carbides and hydrides, but the aging-overaging cycle is too great to be defined in these terms alone. At present the response at 200 C is not explainable.

The response of the titanium-copper binary alloy to heat treatment (Fig. 7) is comparable to the results of prior investigators over the range of times and temperatures studied.

The response of the titanium-copper-cerium alloy is quite different from the copper binary. Appreciable hardening is experienced at all temperatures. Maximum hardness is achieved in 2 hr at 400 C. Peak hardnesses at higher and lower temperatures occur at approximately the same time but are always smaller. The only exception is 600 C where peak hardness occurs at less than 30 min. The hardness results are plotted in Fig. 8.

The results of precipitation treatments of the titanium-silver binary show a hardening response to occur at temperatures above 300 C. At 200 and 300 C there appears to be a very slight hardening. At 400 and 500 C appreciable hardening occurs, with a maximum hardness attained after 3 hr at 400 C. At 600 C the hardness increase is very small. The response of this alloy is characterized by very rapid aging and overaging. In the time range 30 min to 1 hr, a softening occurs at all temperatures. The results of precipitation-hardening treatments on the titanium-silver binary are plotted in Fig. 9.

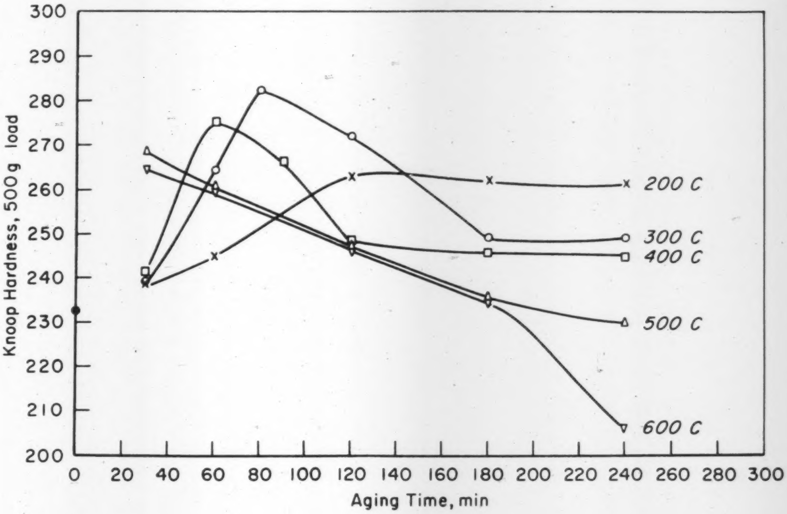

Fig. 10.—Precipitation Hardening Response of Titanium-Silver-Cerium Alloy at Various Temperatures.

The effect of the cerium addition on the hardness response of the titanium-silver alloy is to move the precipitation reaction to lower temperatures and shorter times while effecting a slight increase in maximum hardness. The titanium-silver-cerium alloy shows a hardening at all temperatures, with maximum hardness occurring in $1\frac{1}{2}$ hr at 300 C. The characteristic rapid aging and overaging of the silver binary is also found in the silver-cerium ternary at all temperatures except 200 C where a hardness plateau is experienced beyond peak hardness. The results for the titanium-silver-cerium alloy are plotted in Fig. 10.

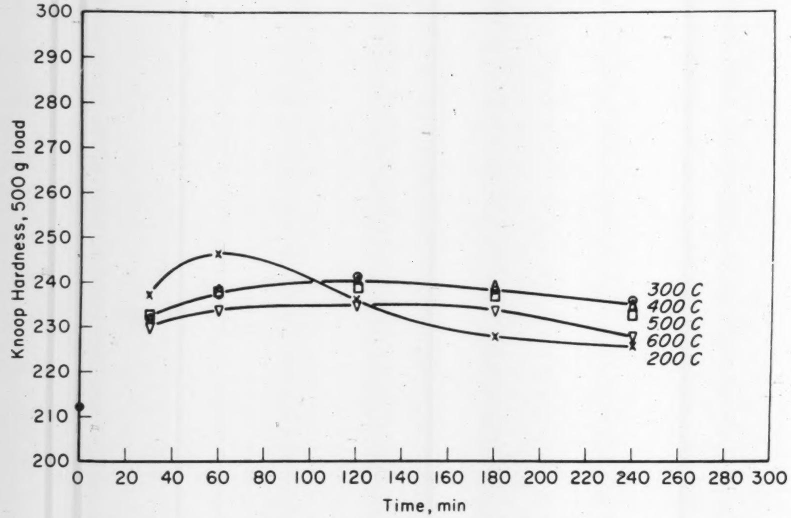

FIG. 11.—Precipitation Hardening Response of Titanium-Silicon Alloy at Various Temperatures.

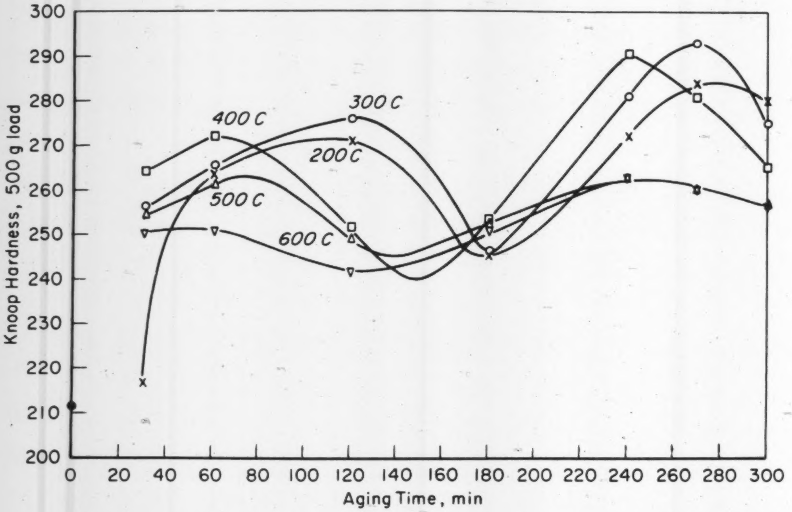

FIG. 12.—Precipitation Hardening Response of Titanium-Silicon-Cerium Alloy at Various Temperatures.

Precipitation-hardening treatments of the titanium-silicon binary alloy resulted in very shallow hardening at all temperatures. The results are similar to those of Antes and Edelman (6). Hardening occurring at 200 C was attributed to carbide and hydride precipitation rather than to silicide precipitation. The results for this alloy are plotted in Fig. 11.

Addition of cerium to the titanium-silicon alloy produces very different results than observed in the other alloys. A double hardness peak is produced as shown in Fig. 12. The first peak occurs in the range of 1 to 2 hr, and the second peak occurs in the range 4 to 4½ hr, depending on temperature. Maximum hardness on both sets of peaks is achieved by aging at 300 C, 2 hr for the first peak, and 3½ hr for the second peak. The hardness curves for the ternary titanium-cerium-silicon alloy (Fig. 12) bear no resemblance to those for the titanium-silicon binary alloy, nor can they be explained in terms of a combination of results from the titanium-cerium and titanium-silicon binaries.

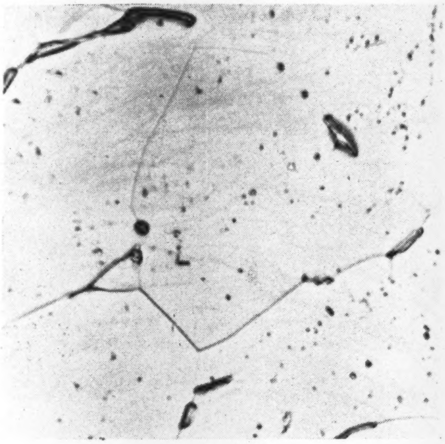

Fig. 13.—Microstructure of Titanium-Cerium Alloy Aged at 500 C for 170 hr. Equiaxed alpha grains with Ce at boundaries and a dispersion of $CeO_2$ within the grains (× 1000).

*Microstructure of Alloys:*

The microstructures of the pure titanium consisted of uniform equiaxed grains of alpha titanium. The microstructures of the titanium-silver, titanium-copper, and titanium-silicon binaries were identical except for the presence of small spheroidal particles randomly distributed throughout the matrix. The microstructure of the titanium-cerium alloy differed from the other binaries. It too consisted of equiaxed alpha grains plus a random precipitate, but there was also an agglomerated second phase present which was concentrated at grain boundaries. Due to the prolonged time at aging temperature of the microspecimen, it appears reasonable to identify the fine dispersion as $CeO_2$ and the agglomerated phase as cerium. Figure 13 is typical of the microstructure of this alloy. The structure of the titanium-copper-cerium alloy (Fig. 14) consisted of an alpha matrix plus a dispersed phase. The grain structure, however, was not equiaxed but elongated, with the precipitated phase outlining a faint lamellar pattern. It was doubtful that the alloy was not recrystallized, but to ascertain recrystallization, X-ray diffraction pat-

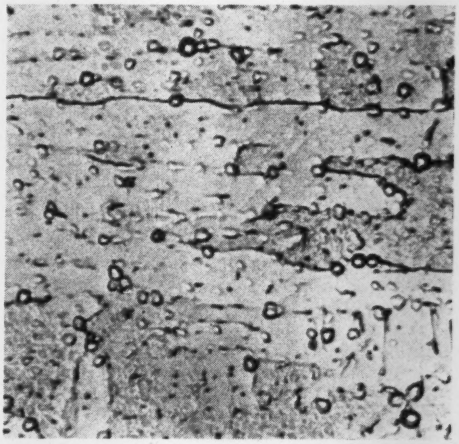

Fig. 14.—Microstructure of Titanium-Copper-Cerium Alloy Aged at 500 C for 170 hr. Elongated alpha grains with a mixture of $CeO_2$ and an unidentified phase dispersed throughout the matrix (× 1000).

terns were obtained for the material. The intensity of the (0002) reflection was found to be very small in the diffraction pattern. If the material was still cold-worked the basal plane would be perpendicular to the direction of applied force, that is, parallel to the surface and thus the (0002) reflection would be strong. The presence of the elongated grain structure is unexplainable, since the control sample consisted of equiaxed alpha grains. It is possible that a slight difference in furnace temperature might cause recrystallization to be parasitic rather than normal. The dispersed phase in this alloy was unidentifiable by metallographic means. The titanium-silver-cerium alloy had a microstructure similar to that of the binary alloys: an equiaxed alpha matrix plus a spheroidal dispersed phase (Fig. 15). The particle size of the second phase varies considerably, with relatively coarse particles at grain boundaries and finer particles within the grains. Identification of the second phase in this alloy was also impossible by metallographic means.

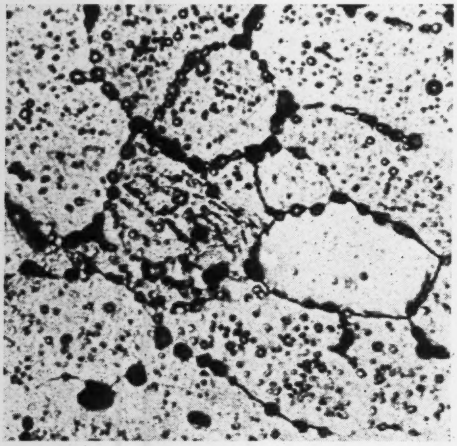

FIG. 15.—Microstructure of Titanium-Silver-Cerium Alloy Aged at 500 C for 170 hr. Equiaxed alpha grains with a mixture of $CeO_2$ and an unidentified phase at grain boundaries and within the grain (× 1000).

For the titanium-silicon-cerium alloy, the microstructure (Fig. 16), like that of the titanium-copper-cerium alloy, consisted of elongated alpha grains plus a dispersed phase of spheroidal and agglomerated particles. The agglomerated particles are similar to those of cerium in the titanium-cerium binary and are thus tentatively identified as cerium.

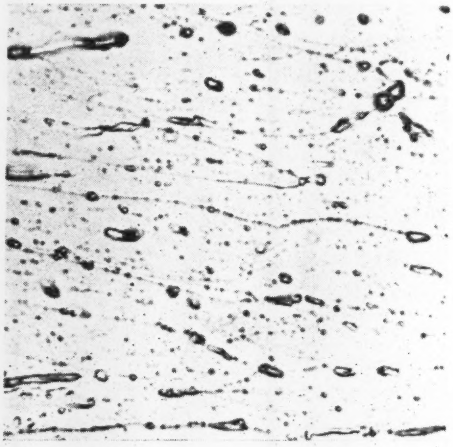

FIG. 16.—Microstructure of Titanium-Silicon-Cerium Alloy Aged at 500 C for 170 hr. Elongated alpha grains with Ce, $CeO_2$, and possibly a third phase at grain boundaries and within the grains (× 1000).

*Phase Identification in Alloys:*

X-ray patterns of the residues of brominated samples of all the alloys contained lines for $TiBr_4$ and titanium. This was due to incomplete reaction during bromination. Lines for $CeO_2$ were found in all alloys containing cerium.

In the patterns for the binary alloys, lines other than those stated above were for the phases predictable from the phase diagram. The lines in the cerium binary were identified with $CeBr_3$, which would be the reaction product of cerium and bromine vapor.

The patterns of the ternary alloys were more complicated. In the titanium-copper-cerium alloy the pattern contained the ever-present lines for titanium, $TiBr_4$, and $CeO_2$ plus very faint traces of the strong lines of $Ti_2Cu$ and a stronger set of lines not identifiable with any known compound. The *d* spacings for these lines are given in Table II. Tentative identification of the compound was made based on the assumptions that: (1) The compound is formed of two face-centered cubic metals and thus should be face-centered cubic itself; (2) The $a_0$ of the compound should fall between the $a_0$ values for copper and cerium. Indices of the characteristic lines of a face-centered cubic metal were assigned to the unknown lines, setting the line with the largest *d* spacing of the (111). The resultant $a_0$ value of 4.385 Å was taken as the true lattice constant value of the phase. This value is almost a mean value of the lattice constants of cerium and copper and indicates a phase composed of almost equal amounts of each element.

TABLE II.—*d* VALUES FOR UNIDENTIFIED LINES IN X-RAY PATTERNS.

| Alloy | *d* | Indices | Alloy | *d* | Indices |
|---|---|---|---|---|---|
| Titanium-Copper-Cerium | 2.47 | (111) | Titanium-Silicon-Cerium | 2.23 | (111) |
| | 1.46 | (200) | | 1.87 | (200) |
| | 1.37 | (220) | | 1.66 | (220) |
| | 1.14 | (311) | | 1.29 | (311) |
| | 1.09 | (222) | | 1.02 | (222) |
| Titanium-Silicon-Cerium | 1.37 | ... | | 0.92 | (400) |
| | 1.19 | ... | | 0.87 | (331) |
| | 0.89 | ... | | 0.83 | (420) |
| | 0.86 | ... | | | |

The diffraction pattern from the bromine residue of the titanium-cerium-silver alloy was similar to that from the titanium-cerium-copper alloy. It was composed of lines from titanium, $TiBr_4$, and $CeO_2$, plus lines of TiAg and a set of unknown lines. Using the same method as stated above, the $a_0$ value for this set of unknown lines is found to be 4.094 Å.

The X-ray pattern from the bromine residue of the titanium-silicon-cerium alloy contained lines for titanium, $TiBr_4$, and $CeO_2$ only. This residue was further reacted with bromine to eliminate all the titanium. The X-ray pattern from the final residue yielded lines for $TiO_2$, $CeO_2$ and $CeBr_3$, plus five other lines (see Table II). One of the five unidentified lines has the same *d* spacing as the 0002) plane of $Ti_5Si_3$. Since this line is the strongest reflection of the $Ti_5Si_3$ pattern, it was identified as the (0002) plane of this compound and indicates that a very small amount of $Ti_5Si_3$ is present. The $CeBr_3$ lines show elemental cerium to be present in the aged alloy.

## CONCLUSIONS

The results of the precipitation hardening study show that the original hope, that of obtaining a precipitation hardenable alloy of titanium with cerium without the precipitation of elemental cerium, has been realized in the case of the titanium-copper-cerium and the titanium-silver-cerium alloys. The titanium-copper-cerium alloy appears to hold the most promise for commercial application. A greater hardness is attainable, with a slower overaging reaction than in the titanium-silver-cerium alloy.

The double peak of the titanium-silver-cerium alloy is not explainable as yet. In any case the alloy does not have much potential for commercial application due to the presence of elemental cerium in the aged material.

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## REFERENCES

(1) F. C. Holden, A. A. Watts, H. R. Ogden, and R. I. Jaffee, "Heat Treatment and Mechanical Properties of Ti-Cu Alloys," *Journal of Metais*, Vol. 7, No. 1, Section 2, Jan. 1955, pp. 117–125.

(2) L. M. Howe, E. Saarema, and J. B. Parr, "Precipitation Hardening in a Ti-Cu Alloy," *Journal of Metals*, Vol. 8, No. 5, Section 2, May, 1956, pp. 512–513.

(3) R. J. Van Thyne and H. D. Kessler, "Brazing and Soldering of Titanium and Titanium Alloys," *Final Technical Report*, Watertown Arsenal, DA-11-O22-ORD-288 Jan. 1953, pp. 59–63.

(4) H. K. Adenstadt and W. R. Freeman, "The Tentative Titanium-Silver Binary System," *Command Technical Report No. 53-109*, Wright Air Development Center, Apr. 1953.

(5) H. W. Worner, "The Structure of Titanium-Silver Alloys in the Range 0–30 Atomic Per Cent Silver," *Journal*, Institute of Metals, Vol. 82, pp 222–227, (1953–1954).

(6) H. W. Antes and E. A. Edelman, unpublished research on titanium-silicon alloys. Frankford Arsenal.

(7) J. L. Taylor, "Further Observations on the Effects of Cerium Additions on Titanium and Some Commercial Titanium Alloys," Physical Metallurgy Symposium of the Metallurgical Advisory Committee on Titanium, Watertown Arsenal, Sept. 1955, pp. 56–57.

(8) J. L. Taylor, "Preliminary Investigation of the Ti-Ce System," *Journal of Metals*, Vol. 9, No. 1, Section 2, Jan. 1957, pp. 94–96.

(9) R. H. Hiltz and F. W. Cotter, "Cerium in Ti-6% Al-4% V Alloys," Physical Metallurgy Symposium of the Metallurgical Advisory Committee on Titanium, Case Institute of Technology, Nov. 1956, p. 54.

(10) W. Trzebiatowski, J. Berak, and T. Romotowski. "The Structure of Titanium-Copper and Titanium-Tungsten Alloys," *Roczniki Chemii*, Vol. 25, pp. 516–517, Brutcher Translation No. 2990, May 1951 and 1953.

(11) M. Hansen, H. D. Kessler, and D. J. McPherson, "The Titanium-Silicon System," *Transactions*, Am. Soc. Metals, Vol. 44, pp. 518–538 (1952).

# EMBRITTLING TENDENCIES OF AUSTENITIC SUPERHEATER MATERIALS AT ELEVATED TEMPERATURES*

By J. Hoke,[1] F. Eberle,[2] and R. D. Wylie[3]

## Synopsis

To determine more fully the effect of long-time elevated-temperature exposure on the properties of standard as well as developmental austenitic materials, nine wrought and nine weld alloys were exposed in the temperature range of 1200 to 1500 F for periods of time up to 20,000 hr. Changes in the properties are reported principally by room- and elevated-temperature notched-bar impact tests as well as by tension and side-bend tests.

All materials investigated suffered deterioration of the impact properties to some extent after aging. However, most wrought materials would appear to have adequate impact strength remaining for superheater service, even after aging for long periods of time. Most weld metals were seriously embrittled by aging, with those least affected being 16 chromium-8 nickel-2 molybdenum, 10 chromium-50 nickel, Inconel, and type 304. Solution annealing after welding lessens such tendencies. Elevated-temperature impact tests indicate greater impact strength at operating than at room temperature after aging.

The increase in steam and metal temperatures in the power industry have led to the use of standard austenitic materials and the development of new alloys for superheater service. Comprehensive information on the effects of long-time high-temperature exposure, such as encountered in superheater service for the generation of steam at 1050 F and above with metal temperatures of 1200 F and above, on the mechanical properties of such austenitic wrought and weld metals is scanty. This has resulted in a certain amount of reluctance to use them, even though such temperature ranges are becoming increasingly common in the trend toward higher steam temperatures and pressures for the more efficient generation of electrical energy. It is known that certain structural changes such as carbide precipitation and sigma formation occur at temperatures in the range encountered in superheater service, which are reflected as changes in the mechanical properties. Those properties most seriously affected are impact resistance and ductility, which are important because of the possibilities of thermal and mechani-

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TABLE I.—CHEMICAL ANALYSES, PER CENT, AND INITIAL CONDITIONS OF EXPERIMENTAL MATERIALS.

| Material | Carbon | Manganese | Silicon | Chromium | Nickel | Molybdenum | Phosphorus | Sulfur | Copper | Others | Initial Condition[a] |
|---|---|---|---|---|---|---|---|---|---|---|---|
| A. WROUGHT MATERIALS | | | | | | | | | | | |
| AISI 304(A) | 0.057 | 0.58 | 0.21 | 18.64 | 10.41 | 0.27 | 0.017 | 0.010 | 0.13 | | ST—1950 F, ½ hr, WQ |
| 304(B) | 0.075 | 1.71 | 0.38 | 18.46 | 10.90 | ... | ... | ... | ... | | ST—1950 F, 1 hr, AC |
| 309 | 0.095 | 1.35 | 0.38 | 21.59 | 14.00 | ... | 0.014 | 0.012 | ... | | ST—2050 F, 1 hr, AC |
| 310 | 0.17 | 1.93 | 0.62 | 25.16 | 20.20 | ... | 0.013 | 0.008 | ... | | ST—2050 F, 1 hr, AC |
| 316(A) | 0.041 | 1.66 | 0.45 | 16.33 | 13.54 | 2.11 | 0.020 | 0.008 | 0.05 | | ST—2000 F, ½ hr, WQ |
| 316(B) | 0.06 | 1.52 | 0.43 | 17.35 | 12.06 | 2.08 | 0.015 | 0.025 | ... | | AR—As received—Hot-rolled + Mill annealed |
| 316(C) | 0.062 | 1.65 | 0.35 | 16.62 | 13.16 | 2.30 | 0.012 | 0.008 | 0.017 | | ST—1950 F, 1 hr, WQ |
| 321 | 0.036 | 1.69 | 0.43 | 17.67 | 12.20 | 0.19 | 0.016 | 0.011 | 0.14 | 0.47 titanium | ST—1950 F, ½ hr, WQ |
| 347 | 0.055 | 1.59 | 0.43 | 18.02 | 10.68 | ... | 0.014 | 0.020 | ... | 0.70 columbium | ST—1950 F, 1 hr, AC |
| 16-8-2 | 0.07 | 1.43 | 0.35 | 16.16 | 8.65 | 1.60 | ... | ... | ... | | ST—1950 F, 1 hr, WQ |
| 15-15N | 0.13 | 1.19 | 0.26 | 15.46 | 13.89 | 1.15 | ... | ... | ... | 1.62 columbium, 0.065 tantalum | ST—2250 F, ½ hr, WQ |
| Armco 17-14 copper, molybdenum | 0.086 | 0.78 | 0.53 | 16.02 | 14.33 | 2.43 | 0.025 | 0.012 | 3.03 | | ST—2250 F, ½ hr, WQ |
| B. WELD METALS | | | | | | | | | | | |
| AISI 304 | 0.10 | 1.55 | 0.22 | 19.78 | 10.04 | ... | 0.039 | 0.011 | ... | | AW—As welded |
| 316 | 0.10 | 1.99 | 0.22 | 18.13 | 11.91 | 2.00 | 0.037 | 0.010 | ... | | AW—As welded |
| 347(A) | 0.10 | 1.92 | 0.17 | 19.85 | 9.58 | 0.13 | 0.042 | 0.010 | ... | 0.78 columbium | AW—As welded |
| 347(B) | 0.10 | 2.30 | 0.44 | 19.09 | 9.25 | ... | ... | ... | ... | 1.20 columbium | (1) AW—As welded<br>(2) AW + ST—1950 F, 1 hr, AC |
| 19-9 tungsten, molybdenum | 0.13 | 1.39 | 0.35 | 19.50 | 8.55 | 0.56 | 0.045 | 0.011 | ... | 1.87 columbium, 1.07 tungsten | AW—As welded |
| 16-8-2 | 0.086 | 1.65 | 0.45 | 15.87 | 8.37 | 1.42 | 0.014 | 0.020 | ... | | (1) AW—As welded<br>(2) AW + ST—1950 F, 1 hr, AC |
| Inconel | 0.063 | 0.91 | 0.41 | 15.34 | 73.60 | ... | 0.015 | 0.012 | ... | 2.11 columbium | AW—As welded |
| Armco 17-14 copper, molybdenum | 0.15 | 1.27 | 0.53 | 16.45 | 14.15 | 2.45 | 0.034 | 0.022 | 3.10 | 0.90 columbium | AW—As welded |
| 10 chromium-50 nickel | ... | ... | ... | 10.0 | 50.0 | ... | ... | ... | ... | | AW—As welded |
| 10 chromium-50 nickel, columbium, tantalum | ... | ... | ... | 10.0 | 50.0 | ... | ... | ... | ... | 2.5 columbium, tantalum | AW—As welded |

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[a] ST = Solution-treated.
WQ = Water-quenched.
AC = Air cooled.

cal shock and the need for spreading of tubes for maintenance purposes.

Wilder and coworkers (**1**, **2**)[4] are in the midst of a very extensive program for the evaluation of the stability of steels at elevated temperatures, which includes several of the austenitic stainless materials. The work is a comprehensive study of weld and parent metal microstructures, tensile and creep properties, as well as impact, hardness, and oxidation characteristics at temperatures from 900 to 1200 F. Lowered impact strengths of types 304 and 316 materials are attributed to carbide precipitation, and in types 321 and 347, to sigma formation. Payson (**3**) has found that there is an appreciable drop in the notch impact resistance of several of the austenitic stainless steels after only 15 hr of heating at 1400 and 1600 F, particularly in those with higher silicon contents. Test results on certain austenitic materials after exposure to actual superheater operating conditions have been reported by Hoke and Eberle (**4**). The property most notably affected was impact strength, with changes being most rapid during the earlier part of exposure, followed by a lower rate of change thereafter.

Work has been in progress within the author's company during the past several years, investigating the effects of long-time aging at elevated temperatures on various properties of standard and developmental austenitic superheater materials. These were evaluated by tensile, hardness, impact, and bend data. Changes in the microstructures with aging were followed metallographically. Attention is concentrated in this report on changes in the impact strength, since it appears that this property is most seriously affected by aging.

## MATERIALS AND TEST PROCEDURES

Test results for nine wrought and nine weld austenitic alloys are presented in this report. Certain of these are standard materials developed originally for corrosion resistance in aqueous media and subsequently employed for high-temperature service; others have been developed more recently and are being evaluated as to their possible improvement over existing alloys. Chemical compositions and initial conditions of these various wrought and weld materials are given in Table I. The 17 chromium-14 nickel (with copper and molybdenum) and the 15 chromium-15 nickel materials have been developed for improved elevated-temperature-strength properties. The 16 chromium-8 nickel-2 molybdenum was developed to minimize weld embrittlement tendencies and is now also being used as a wrought high-temperature material. The 10 chromium-50 nickel compositions were developed as low-expansion electrode materials for joining ferritic steels to austenitic alloys, and Inconel is also used for this application.

The experimental material for weld metal impact and bend specimens was prepared as butt-welded plate, $\frac{5}{8}$ in. thick with a long weld in the center. Welding was done manually with direct current - reverse polarity, in the flat position, using $\frac{1}{8}$-in. electrodes with a maximum of 250 F preheat to remove chill and without postheat treatment. Material for the weld metal tension tests was deposited as all-weld-metal pads. Specimens for base metal properties were obtained from plate materials.

Aging treatments were carried out in electrically heated muffle furnaces at various temperatures from 1200 to 1500 F for periods of time up to 20,000 hr. Periodically during these aging treatments, material was removed for test

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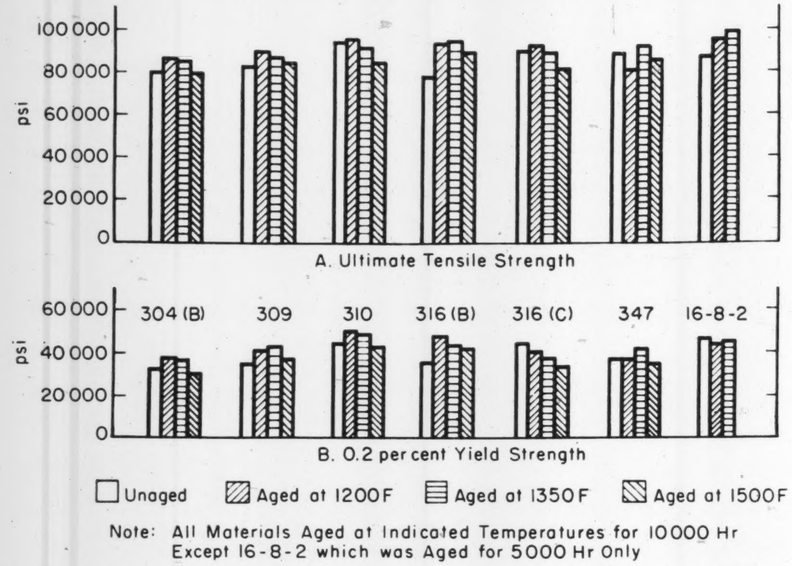
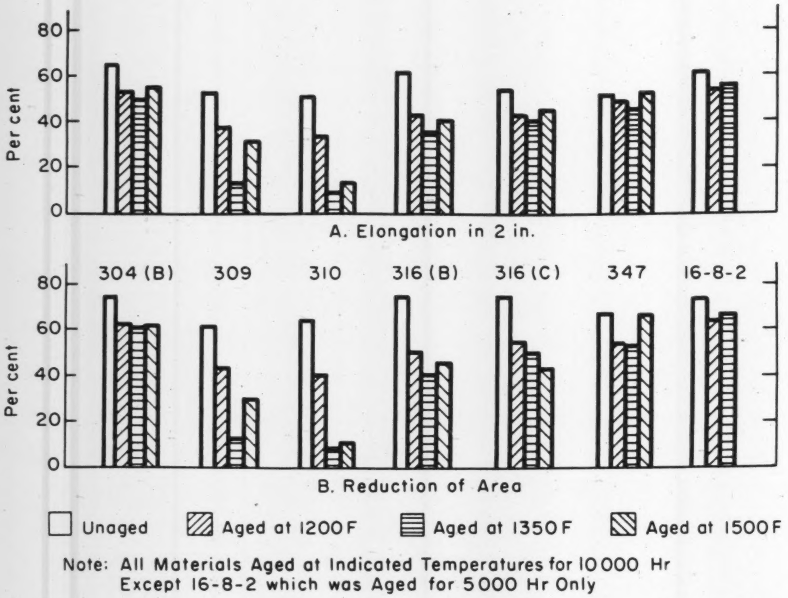
[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 831.



Note: All Materials Aged at Indicated Temperatures for 10 000 Hr
Except 16-8-2 which was Aged for 5000 Hr Only

FIG. 1.—Effect of Aging on Room-Temperature Tensile Strength Properties of Wrought Materials.



Note: All Materials Aged at Indicated Temperatures for 10 000 Hr
Except 16-8-2 which was Aged for 5000 Hr Only

FIG. 2.—Effect of Aging on Room-Temperature Tensile Ductility Properties of Wrought Materials.

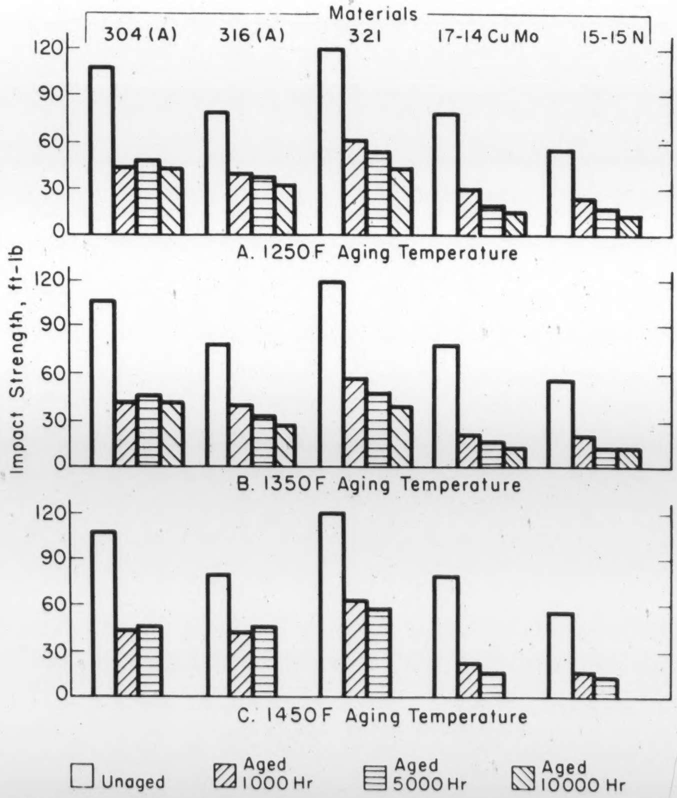


FIG. 3.—Effect of Aging on Room-Temperature Charpy Keyhole Impact Strength Values of Wrought Materials.

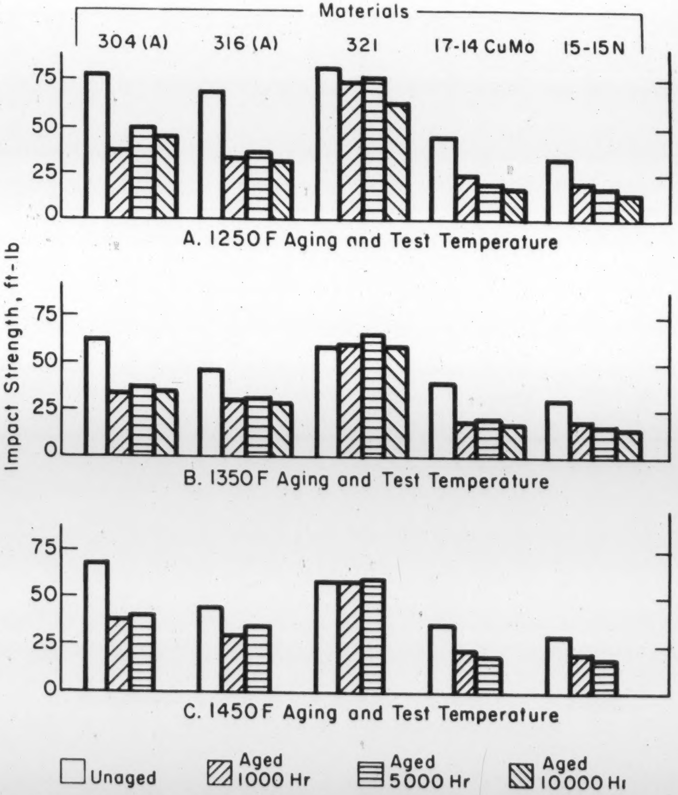


FIG. 4.—Effect of Aging on Elevated-Temperature Charpy Keyhole Impact Strength Values of Wrought Materials.

purposes in order that the progress of change during aging could be followed.

Changes in shock resistance reported were determined by Charpy notched-bar impact tests at room and aging temperatures, in strength properties by tension tests, and in ductility by guided side-bend and tension tests. Impact values reported are an average of three or four tests. The guided side-bend tests were carried out on specimens $\frac{5}{8}$ by $\frac{3}{8}$ in. in cross-section.

## PRESENTATION AND DISCUSSION OF RESULTS

Test results are reported graphically insofar as possible, with particular emphasis on impact data. Tension and bend test results are included for general interest.

*Tension Tests:*

Effects of aging at 1200, 1350, and 1500 F on the room-temperature tensile properties of seven wrought materials are shown in Figs. 1 and 2. Strength and ductility values are reported in the unaged condition and after aging for 10,000 hr, except for the 16 chromium-8 nickel-2 molybdenum material which has only accumulated 5000 hr aging time.

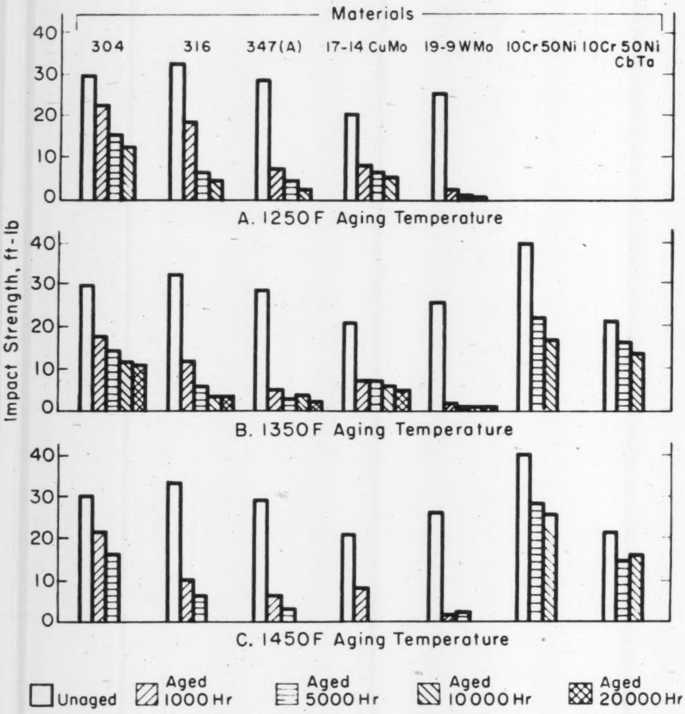


FIG. 5.—Effect of Aging on Room-Temperature Charpy Keyhole Impact Strength of Weld Metals.

These results indicate that aging does not markedly affect the strength, properties, as reflected by the ultimate tensile and 0.2 per cent yield strength values. The effect on ductility properties was more pronounced, as shown by the resulting lower elongation and reduction-of-area values.

*Impact Tests:*

The effects of aging on room-temperature and aging-temperature Charpy "keyhole" impact strength values of five wrought materials aged at 1250, 1350, and 1450 F are shown in Figs. 3 and 4. All materials suffered a marked loss in room-temperature toughness after aging at the elevated temperatures, but none of the values fall below 10-ft-lb. This embrittling effect was less pronounced for the elevated-temperature impact tests. At elevated testing temperatures, the values are not as low in most cases after aging as at room temperature, and the percentage decrease is less because the unaged materials have lower initial impact strength. In general, the most rapid decrease in impact strength occurred during the first 1000 hr, with a decreasing rate of change thereafter. Type 321 showed very little change in elevated-temperature impact strength after aging.

The room- and elevated-temperature impact test results obtained with keyhole specimens for seven weld metals are graphically illustrated in Figs. 5 and 6. Here it is observed that these materials experienced a much more drastic loss in room-temperature impact resistance than the wrought alloys. Those least affected were the two 10 chromium-50 nickel-type alloys and non-stabilized 18-8 type 304. Types 316 and 347, 17 chromium-14 nickel (with copper and molybdenum), and 19 chromium-9 nickel (with tungsten and molybdenum) were rather seriously embrittled after ex-

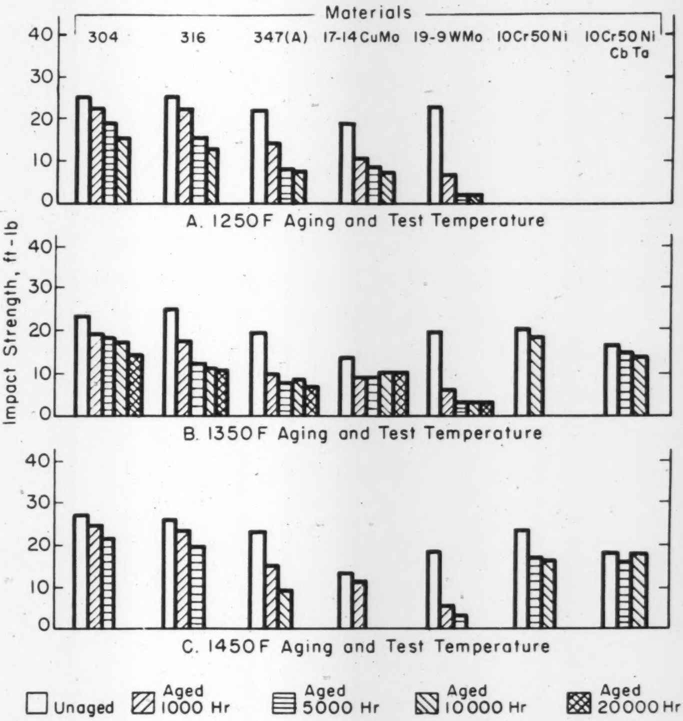


FIG. 6.—Effect of Aging on Elevated-Temperature Charpy Keyhole Impact Strength of Weld Metals.

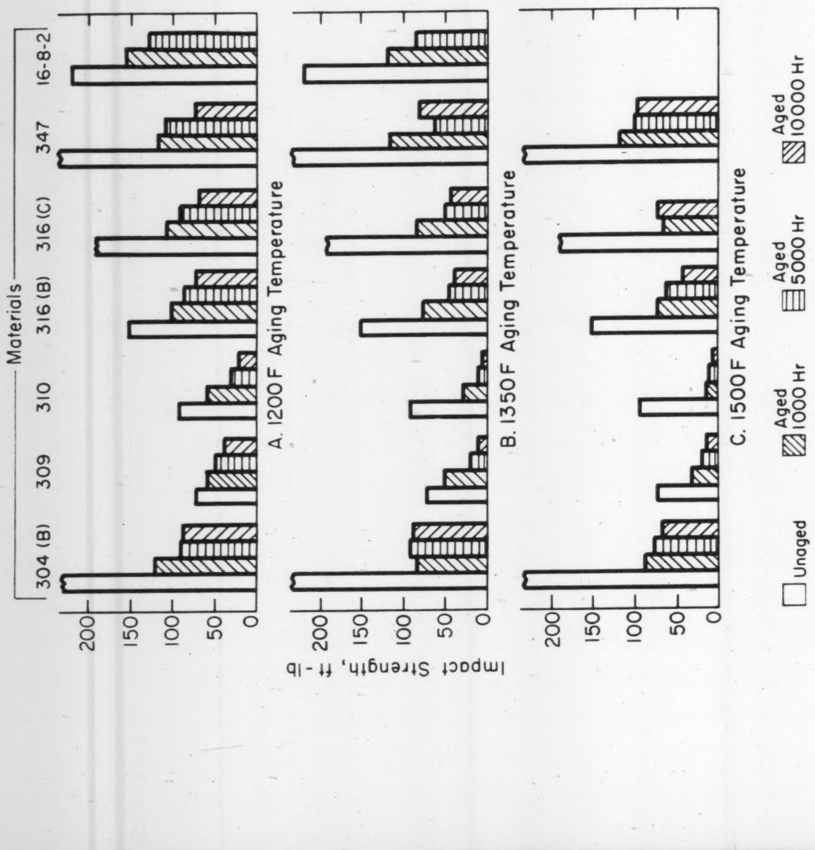


FIG. 7.—Effect of Aging on Room-Temperature Charpy V-Notch Impact Strength of Wrought Materials.

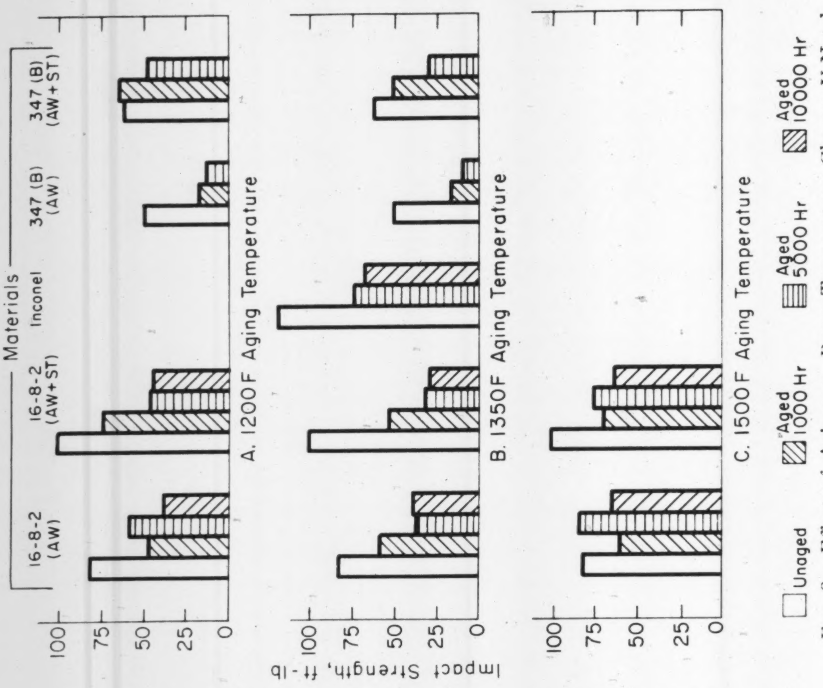


FIG. 8.—Effect of Aging on Room-Temperature Charpy V-Notch Impact Strength of Weld Metals.

tended aging. The elevated-temperature impact strengths of the weld deposits after aging are slightly higher than those for room temperature. Again the most promising materials were the 10 chromium-50 nickel alloys and type 304. When comparing the effects of aging temperatures, it appears that none of the materials is particularly sensitive to embrittlement at any one of the three test temperatures.

The effects of aging on room-temperature Charpy V-notch strengths of seven wrought and three weld materials are shown in Figs. 7 and 8. Again a marked decrease in values is noted for all materials. Of particular interest is the decreased embrittling tendency of the low-ferrite type 347 material if given a solution treatment after aging. It should be emphasized that, in comparing the various impact values in this report, Charpy keyhole and V-notch results do not duplicate, and the results are not directly comparable.

The results of the aging tests indicate that, based on an arbitrary acceptance level of 10 ft-lb, all of the wrought materials tested, except types 309 and 310, maintain sufficient notch toughness. The fact that type 310 has been used successfully in superheaters and chemical process equipment suggests that the lowest acceptable impact strength is not known for service where no impact-type loads are to be expected. A similar pattern exists for weld deposits and, in addition, progress has been made in developing

TABLE II.—EFFECT OF AGING ON WELD METAL BEND TESTING RESULTS.

BEND DATA AFTER AGING

| Material[a] | Aging Temp, deg Fahr | 0 hr | | 1000 hr | | 5000 hr | | 10 000 hr | | 20 000 hr | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Bend Angle | Failure[b] | Bend Angle | Failure[b] | Bend Angle | Failure[b] | Bend Angle | Failure[b] | Bend Angle | Failure[b] |
| 304 | 1250 | 180, 180 | 1, 1 | 180, 180 | 1, 1 | 180, 180 | 1, 1 | 180, 180 | 1, 1 | ... | ... |
| | 1350 | 180, 180 | 1, 1 | 180, 180 | 1, 1 | 180, 90 | 1, 3 | 180, 180 | 1, 1 | 162 | 2 |
| | 1450 | 180, 180 | 1, 1 | 180 | 1 | 180, 180 | 1, 1 | ... | ... | ... | ... |
| 316 | 1250 | 180, 180 | 1, 1 | 180, 180 | 1, 1 | 180 | 1 | 44, 55 | 4, 4 | ... | ... |
| | 1350 | 180, 180 | 1, 1 | 180, 180 | 1, 1 | 180, 55 | 1, 4 | 54, 51 | 4, 4 | 41 | 5 |
| | 1450 | 180, 180 | 1, 1 | 180, 141 | 1, 3 | 180, 59 | 1, 4 | ... | ... | ... | ... |
| 347(A) | 1250 | 180, 180 | 1, 1 | 132, 53 | 2, 2 | 17, 93 | 3, 2 | 54, 23 | 2, 3 | ... | ... |
| | 1350 | 180, 180 | 1, 1 | 180, 180 | 1, 1 | 61, 63 | 6, 6 | 180, 180 | 1, 1 | 58 | 5 |
| | 1450 | 180, 180 | 1, 1 | 89, 26 | 2, 2 | 180, 80 | 1, 3 | ... | ... | ... | ... |
| 17-14 copper, molybdenum | 1250 | 180, 180 | 1, 1 | 34, 28 | 3, 3 | 31, 53 | 2, 5 | 64, 10 | 5, 3 | ... | ... |
| | 1350 | 180, 180 | 1, 1 | 20, 42 | 3, 3 | 44, 41 | 4, 4 | 52, 11 | 3, 3 | 58 | 5 |
| | 1450 | 180, 180 | 1, 1 | 180, 50 | 1, 2 | 52, 35 | 4, 3 | ... | ... | ... | ... |
| 19-9 tungsten, molybdenum | 1250 | 180, 180 | 1, 1 | 50, 21 | 5, 5 | 18, 11 | 6, 6 | 14, 16 | 5, 5 | ... | ... |
| | 1350 | 180, 180 | 1, 1 | 46, 40 | 5, 5 | 15, 21 | 5, 5 | 15, 17 | 5, 5 | 14 | 5 |
| | 1450 | 180, 180 | 1, 1 | 25, 29 | 5, 6 | 16, 25 | 5, 5 | ... | ... | ... | ... |

[a] Initial condition—As welded,
Duplicate tests run where two sets of data included.

[b] Failures classified according to following system:
1—Full 180 deg guided bend, no defects
2—Weld fusion zone tear
3—Weld fusion zone crack
4—Weld metal crack
5—Abrupt weld metal fracture
6—Abrupt fusion zone fracture

more stable materials with considerably reduced embrittling tendencies. Alloys 16 chromium-8 nickel-2 molybdenum, 10 chromium-50 nickel, Inconel, and solution-treated low-ferrite type 347 are examples of this.

*Bend Tests:*

Weld metal guided side-bend test data for five materials are presented in Table II. Evaluation is based on the bend angles the specimens withstood before a significant flaw or failure occurred. A brief description of the failures is also indicated, or if no defects were observed during the test, the specimen is reported to have taken a full 180-deg bend. This test does not appear to be as sensitive to aging effects as the impact test. The 19 chromium-9 nickel (with tungsten and molybdenum) weld metal specimens had very low ductility after aging and failed by fracturing abruptly. Type 304 was practically unaffected according to the bend test results, and types 316 and 347 and 17 chromium-14 nickel (with tungsten and molybdenum) showed a certain amount of deterioration.

*Microstructural Observations:*

Changes in the mechanical properties are reflections of changes in the microstructure of the various alloys. Even though all materials discussed in this report are considered austenitic, certain changes may occur during welding or subsequent aging that produce structures not fully austenitic. Due to compositional gradients, as-deposited weld metals frequently contain an appreciable amount of free ferrite, even though wrought materials of similar compositions would be fully austenitic. It was present in varying amounts for all weld metals investigated except 17 chromium-14 nickel (with copper and molybdenum), 10 chromium-50 nickel, and Inconel. A second unidentified phase was present in the 10 chromium-50 nickel (with columbium and tantalum) alloy as welded, which coalesced slightly upon aging.

Many of the austenitic superheater materials, and in particular weldments, have a tendency to develop sigma phase at the temperatures investigated in this work. The amount and distribution may vary considerably and influence the mechanical properties accordingly. It was observed that after 1000 hr a considerable amount of sigma was already present and no ferrite remained in those weld metals containing it originally except 16 chromium-8 nickel-2 molybdenum. Sigma became very extensive in the 19 chromium-9 nickel (with tungsten and molybdenum) material, which correlates with the very low impact strength developed. Sigma formation was not extensive for most of the wrought materials, except types 309 and 310, as again indicated by the low impact properties. However, it did develop as a discontinuous grain boundary network for types 321 and 347 and for 17 chromium-14 nickel (with copper and molybdenum).

Many of the materials developed extensive precipitates during aging. Those weld metals developing sigma did not have this tendency. The superalloys 17 chromium-14 nickel (with copper and molybdenum) and 15 chromium-15 nickel developed a very heavy general precipitate, and the unstabilized alloys 304 and 316 and 16 chromium-8 nickel-2 molybdenum, both a general and grain-boundary precipitate. A small amount of general precipitate was also observed in the stabilized alloys 321 and 347 and 10 chromium-50 nickel after long aging times.

## Conclusions

1. All materials investigated suffered some deterioration of the room-tempera-

ture impact properties by aging for long periods of time in the temperature range of 1200 to 1500 F.

2. After aging, impact strengths at elevated temperatures tend to be slightly higher than at room temperature.

3. Embrittlement occurs most rapidly during the first 1000 hr or less of exposure, after which the rate of change gradually decreases.

4. All of the wrought materials investigated retained adequate impact strength for superheater service, with the possible exceptions of types 309 and 310.

5. Many of the weld deposits were considerably embrittled; those showing to the best advantage were the 10 chromium-50 nickel alloys, 16 chromium-8 nickel-2 molybdenum, Inconel, type 304, and low-ferrite type 347 in the solution-treated condition.

*Acknowledgment:*

Acknowledgment is made to the various members of the Metallurgical Sections of The Babcock & Wilcox Co. who assisted in this investigation and in particular to F. Snyder, W. Feduska, T. J. Moore, and G. M. Anderson.

## REFERENCES

(1) A. B. Wilder and J. B. Light, "Stability of Steels at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 41, p. 141 (1949).

(2) A. B. Wilder and E. F. Ketterer, "The Structure and Properties of Stainless Steels after Exposure at Elevated Temperatures," Symposium on Strength and Ductility of Metals at Elevated Temperatures, Am. Soc. Testing Mats., p. 237 (1952). (Issued as separate publication *ASTM STP No. 128.*)

(3) P. Payson and C. H. Savage, "Change in Austenitic Chromium-Nickel Steels During Exposure at 1100–1700 F," *Transactions*, Am. Soc. Metals, Vol. 39, p. 404 (1947).

(4) J. Hoke and F. Eberle, "Experimental Superheater for Steam at 2000 psi and 1250 F—Report after 14,281 Hours of Operation," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 79, No. 2, Feb., 1957, p. 307.

# DISCUSSION

MR. A. B. WILDER.[1]—We are particularly interested in this paper because we are conducting a similar investigation at temperatures of 900, 1050 and 1200 F for these alloys. Our tests are not as extensive as those presented by the authors but we are working in this same area. So in reality there is information available then from 900 to 1500 F after long periods of exposure. We expect to expose our alloys up to 100,000 hr and we now have about 75,000 hr exposure.

With regard to the Charpy V-notch and the Charpy keyhole-notch, as the authors pointed out, there should not be a direct comparison made. But I would like to ask a question of whether the same order of embrittlement with regard to the various alloys was observed in the Charpy V-notch as in the Charpy keyhole or whether there were differences there with regard to the order of embrittlement. By that I mean one alloy of a certain type would be embrittled and shown to be embrittled by the keyhole-notch. Would that also be true with the V-notch?

MR. J. H. HOKE (*author*).—The duplicate material data reported here with respect to the two types of impact tests used for evaluation actually came from different projects. Thus, as seen in Table I separate heats of material were investigated. In general though the same trend of decreasing impact strengths with aging time was found by both methods. The tests were not correlated to the extent that details of this nature can be answered conclusively, but it does not appear that the evaluation of materials is dependent upon the type of impact test used.

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[1] Chief Metallurgist, National Tube Division, U. S. Steel Corp., Pittsburgh, Pa.

# INFLUENCE OF NOTCH PREPARATION AND ECCENTRICITY OF LOADING ON THE NOTCH RUPTURE LIFE*

By M. H. Jones,[1] J. L. Shannon, Jr.,[1] and W. F. Brown, Jr.[1]

## Synopsis

The investigation consists of two phases: (1) The influence of notch preparation methods on the rupture life of the chromium-molybdenum-vanadium steel 17-22A(S) and the super alloy A-286; (2) The influence of initial loading eccentricity on the notch rupture life of 17-22A(S). In phase (1), sharp notches were either tool cut or ground before or after complete heat treatment. In phase (2), sharp notches tool cut after heat treatment were used. For both phases testing conditions were selected which yielded high and low notch-rupture strength ratios (notch ductilities).

The results indicate that the 1000 F rupture life of 17-22A(S) is practically independent of the methods of notch preparation investigated. In contrast, the 1200 F rupture life of A-286 was influenced by the method of notch preparation for testing conditions yielding a low notch-rupture strength ratio. Under these conditions notches tool cut after heat treating had the longest lives. The rupture life of notches ground after heat treating was practically equal to notches tool cut or ground before heat treatment. These results for A-286 are compared with similar published data for the super alloy Inconel X-550. Very small eccentricities of loading reduce the notch-rupture strength of 17-22(S) when tested under conditions yielding a low notch-rupture strength ratio. If the testing conditions result in a high notch-rupture strength ratio the influence of small eccentricities is negligible.

It is now generally recognized that any complete evaluation of material properties should include tests on notched specimens. The notch creep-to-rupture test is incorporated in certain AMS specifications[1a] and will undoubtedly be added to other material specifications in the near future. A large and increasing body of literature relates to the influence of notches on the creep-to-rupture properties of various alloys. To date there is no generally accepted method for preparing the notch or testing the specimens.

The present authors have always reported data on tool cut notches, while some investigators (**1, 2, 3, 6**)[2] grind or lap the notch. Furthermore, notch specimens are generally tested using conventional threaded grips and no effective method is

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Lewis Flight Propulsion Laboratory, National Advisory Committee for Aeronautics, Cleveland, Ohio.

[1a] Aeronautical Materials Specifications issued by Society of Automotive Engineers.

[2] The boldface numbers in parentheses refer to a list of references appended to this paper, see p. 849.

TABLE I.—COMPOSITION, HEAT TREATMENT, AND HARDNESS OF ALLOYS TESTED.

| Alloy | Heat Number | Composition, per cent | | | | | | | | | | | Heat Treatment | Rockwell Hardness, C Scale |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Carbon | Chromium | Nickel | Molybdenum | Manganese | Silicon | Aluminum | Vanadium | Titanium | Copper | Iron | | |
| 1722A(S)[a, d] | 31 158 | 0.32 | 1.32 | 0.14 | 0.48 | 0.60 | 0.70 | .... | 0.22 | .... | 0.17 | Balance | 1850 F, ½ hr, air<br>1200 F, 6 hr, air | 35 |
| 17-22A(S)[b, d] | 24 797 | 0.30 | 1.25 | 0.25 | 0.52 | 0.63 | 0.60 | .... | 0.25 | .... | 0.10 | Balance | 1850 F, ½ hr, air<br>1200 F, 6 hr, air | 35 |
| A286[c, e] | 81 227 | 0.054 | 14.57 | 15.22 | 1.23 | .... | .... | 0.21 | 0.29 | 1.98 | .... | Balance | 1800 F, 1 hr, oil<br>1325 F, 16 hr, air | 30 |

[a] Notch preparation tests.

[b] Eccentricity tests.

[c] Arc furnace air melt.

[d] Received as hot rolled, annealed 1 in. diameter bar stock, Rockwell hardness C 18.

[e] Received as solution treated (1650 F, 1 hr, air) ¾ diameter bar stock, Rockwell hardness C 14.

used to control carefully the eccentricity of loading. It is therefore difficult to correlate notch data obtained in different laboratories even for the same specimen geometry.

Regarding notch preparation, it is to be expected that the machining method employed might result in residual stresses and structural changes. Either of these might influence the notch properties to an extent depending on both the material and test conditions. This problem was first recognized by Sachs, Lubahn, and Ebert (**5**) in an investigation of the room temperature notch-tensile properties of SAE 3140. Tool cut notches were wire lapped to final contour and a few were pickled before lapping. However, the results are inconclusive regarding the possible effects of cold work.

More recently Carlson, MacDonald, and Simmons (**1**) reported structural changes at the root of notches ground into stress-rupture specimens of several super alloys. They suggest that the method of notch preparation may influence the rupture strength. Similar observations have been made by Hyler and Simmons (**4**) and by Vitovec and Lazan (**2, 3**) for a number of super alloys. Some of these authors (**3, 4**) describe special lapping and electropolishing techniques designed to remove the disturbed zone. However, none of these investigators present data regarding the influence of notch preparation method on the creep-rupture strength. Voorhees and Freeman (**7, 8**) have shown the notch creep-rupture strength of Inconel X-550 is dependent on the method of notch preparation and suggest the effects observed are primarily due to residual stresses. These very pertinent results will be discussed later.

Regarding the influence of loading eccentricity, a few preliminary results were reported by the present authors (**9**). These show the notch-rupture strength decreases with increasing initial eccentricity for a chromium-molybdenum-vanadium steel tested under conditions

TABLE II.—CONDITIONS OF NOTCH PREPARATION INVESTIGATED.

| Alloy | Machined Condition | Machining Method | Notch Radius, in. | Test Stress, psi |
|---|---|---|---|---|
| 1722A(S) Tested at 1000 F.... | As-received | Tool cut | 0.001<br>0.0085 | 140 000 and<br>60 000 |
| | | Ground | 0.001<br>0.0085 | 140 000 and<br>60 000 |
| | Heat-treated | Tool cut | 0.001<br>0.0085 | 140 000 and<br>60 000 |
| | | Ground | 0.001<br>0.0085 | 140 000 and<br>60 000 |
| A286 Tested at 1200 F........ | As received | Tool cut | 0.001 | 110 000 and<br>54 000 |
| | | Ground | 0.001 | 110 000 and<br>54 000 |
| | Heat-treated | Tool cut | 0.001 | 110 000 and<br>54 000 |
| | | Ground | 0.001 | 110 000 and<br>54 000 |

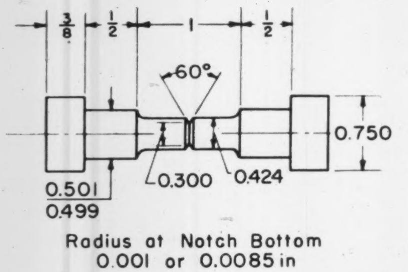
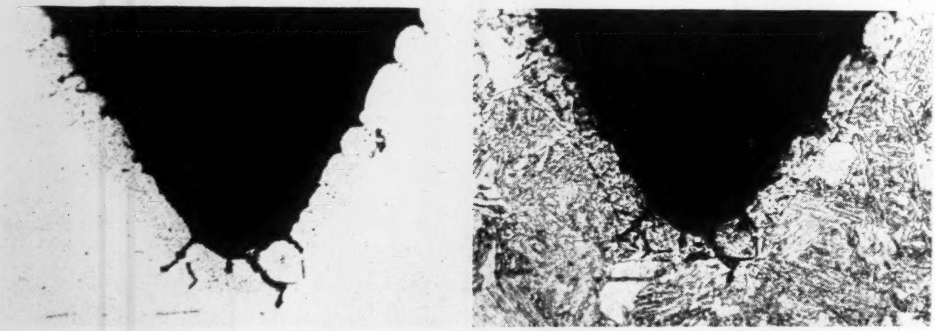


FIG. 1.—Concentric Buttonhead Specimen Used in Investigation of Notch Preparation Methods.



(*a*) Normalized 1850 F for ½ hr in neutral salt bath; unetched.

(*b*) Normalized 1850 F for ½ hr in neutral salt bath; etched Vilella's Martensitic.

FIG. 2.—Photomicrographs of 17-22A(S), 0.001 in. Radius, Notched Specimens (× 750).

yielding a low notch-rupture strength ratio (notch ductility). On the basis of data reported by Sachs, Lubahn, and Ebert (5) for room temperature notch tension tests, it might be expected that the effect of initial eccentricity is a function of the notch strength ratio (notch ductility).

The present investigation is a study of the effect of notch preparation method and eccentricity of loading in creep-to-rupture tests. Specifically an attempt is made to determine: (*a*) the relative strength of ground as compared with tool cut notches for the chromium-molybdenum-vanadium steel 17-22A(S) and the super alloy A-286; (*b*) the possible influence of residual stresses associated with notch machining; (*c*) the influence of loading eccentricity on the notch-rupture strength of 17-22A(S), and (*d*) the effect of the above mentioned variables as influenced by the notch ductility.

## MATERIAL AND PROCEDURE

The chemical composition and heat treatment employed for 17-22A(S) and A-286 are shown in Table I. It will be noted that the heat treatment for 17-22A(S) is not that recommended by the supplier.[3] The normalizing temperature has been raised to 1850 F in order to provide a wide range of notch-rupture strength ratios in reasonable testing times. The heat treatment for A-286 is one commonly recommended by the manufacturer.

### *Specimen Preparation and Heat Treating Techniques:*

The conditions of notch preparation are summarized in Table II and the specimen employed is shown in Fig. 1. It will be noted that particular attention has been given to the problem of concentricity. Specimens were machined with great care, all cylindrical surfaces between the buttonheads being concentric with the notch to within 0.0005 in. The loading surfaces under the buttonheads are flat and square with the longitudinal centerline. In addition, the 0.500-in. diameter is held ±0.001 in. It is this section which provides radial positioning of the specimens with respect to the loading axis.

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[3] Recommended heat treatment for 17-22A (S): 1725 F, ½ hr, air cool; 1200 F, 6 hr, air cool

It will be noted from Table II that notches were either ground or tool cut. Details of these machining methods are given in the Appendix. Two notch radii were investigated for 17-22A(S). The 0.001-in. radius notch corresponds to our general practice and has a sharpness[4] of approximately 150. The 0.0085-in. radius notch has a sharpness of approximately 17 which is the value recommended in certain AMS specifications. Parallel series of tests were made for specimens machined before and after a complete heat treatment.

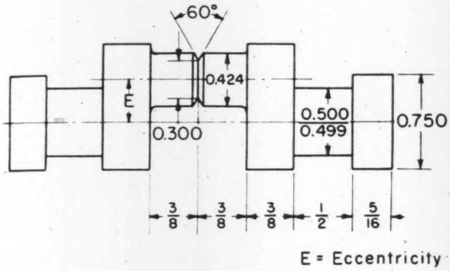

FIG. 3.—Buttonhead Specimen Used to Obtain Various Controlled Amounts of Initial Eccentricity.

Heat treating completely-machined specimens requires considerable care if surface oxidation or decarburization is to be avoided. Initially, attempts were made to heat treat in so-called neutral salts. Several salts containing either rectifiers or special additives were tried using a new aluminum-coated steel pot. Invariably the result was that shown in Fig. 2 for a typical 17-22A(S) tool cut sharp notch. The intergranular attack occurred primarily during normalizing and was not confined to the notch root. The procedure finally adopted was to place the specimen with titanium chips in a plain carbon steel tube having a $\frac{1}{32}$-in. wall and an inside diameter only slightly greater than the head diameter of the specimen. The tube ends were flattened and sealed by welding. All specimens were heat treated in this manner. Those to be heat treated before machining were not notched and had cylindrical dimensions within 0.010 in. of final size. Thus, the cooling rates should be comparable for specimens heat treated before and after machining. This procedure resulted in surfaces which showed no evidence of structural alteration for 17-22A(S). However, A-286 specimens notched before heat treatment exhibit evidence of structural disturbance at the surface which, as will be discussed later is thought to be associated with the heat treating procedure.

Specimens for investigating eccentricity effects, Fig. 3, were also machined with great care. The longitudinal centerline of the test section was offset various controlled amounts from the centerline of the heads. The distance between these two centerlines is defined as the initial eccentricity. All specimens for this investigation were machined from completely heat-treated 1-in. diameter blanks. The 50 per cent notches were tool cut to a radius of 0.001 in.

### *Testing Techniques:*

Specimens for both phases of this investigation were tested in specially designed axial loading creep machines (9). These units are provided with precision loading yokes, buttonhead loading rods and split buttonhead adapters. Using carefully prepared concentric buttonhead specimens, the eccentricities approach the machining tolerances, namely, less than 0.001 in. Such low values cannot be consistently obtained with conventional loading methods employing threaded specimens (**9**). The temperature control circuitry has been previously described (**9**) and yields a maximum deviation of ±3 F from the nominal test temperature. Each specimen was provided with two new thermocouples spot welded approximately $\frac{1}{4}$ in. on either side of the notch. These chromel alumel, asbestos glass-insulated couples were selected to agree with the NBS RP-767 curve within $\pm\frac{1}{5}$ per cent from 600 to 2000 F.

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[4] The notch sharpness is defined as the ratio between one half the diameter at the notched section to the radius at the notch root.

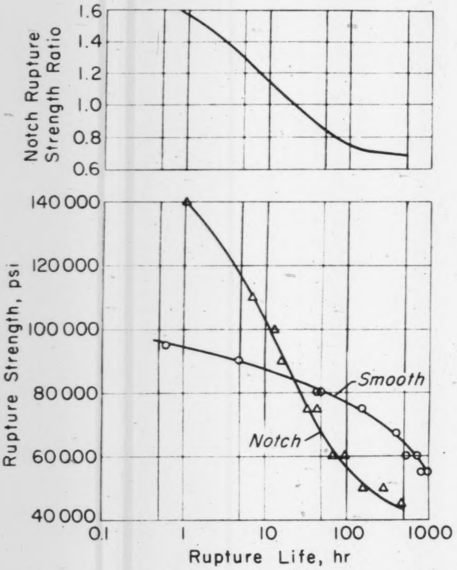

FIG. 4.—1000 F Notch- and Smooth-Rupture Strength and Notch-Rupture Strength Ratio for 17-22A(S) Threaded End Specimens Machined after Heat Treatment (**10**).

*Testing Conditions:*

All tests on 17-22A(S) were carried out at 1000 F. The 1000 F notch and smooth creep-to-rupture strengths and the notch-rupture strenght ratio for this alloy are shown in Fig. 4. These data were taken from a previous publication (**10**)[5] and represent threaded specimens having 50 per cent, 60 deg, 0.001-in. radius notches, tool cut after heat treatment. For the investigation of notch preparation method, stress levels of 140,000 psi and 60,000 psi were selected for each notch radius. The higher stress yields a notch-rupture strength ratio of approximately 1.5, representing a fully notch ductile condition. The lower stress yields a notch-rupture strength ratio of approximately 0.75 and represents a notch brittle condition. For the investigation of eccentricity the same two testing stresses were employed plus a third at 84,000 psi corresponding to a notch-rupture strength ratio of approximately 1.0.

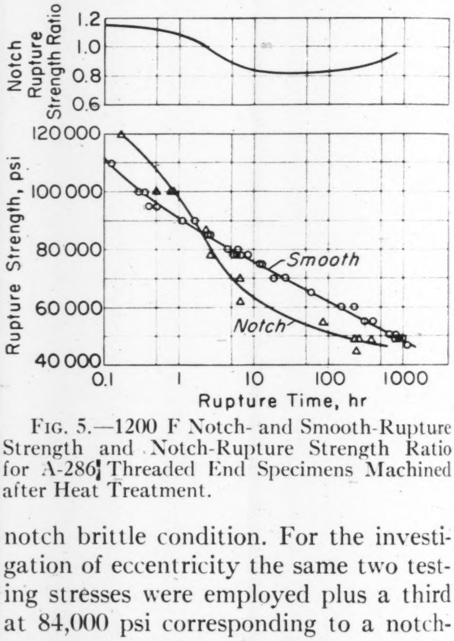

FIG. 5.—1200 F Notch- and Smooth-Rupture Strength and Notch-Rupture Strength Ratio for A-286 Threaded End Specimens Machined after Heat Treatment.

Notch preparation tests on A-286 were conducted at 1200 F at stress levels of 110,000 psi and 54,000 psi. According to Fig. 5 these correspond to notch-rupture strength ratios of approximately 1.15 and 0.80, respectively. The data in Fig. 5 were obtained with threaded

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[5] These data were obtained on a different heat of steel than that used in this investigation. However, the notch strength ratios have been found to be essentially independent of the heat used.

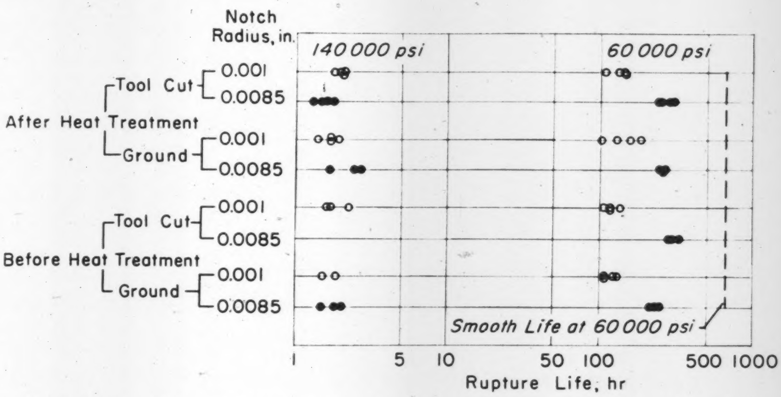

FIG. 6.—1000 F, 140,000 and 60,000 psi. Rupture Life of 17-22A(S) Specimens Notched Before or After Heat Treatment.

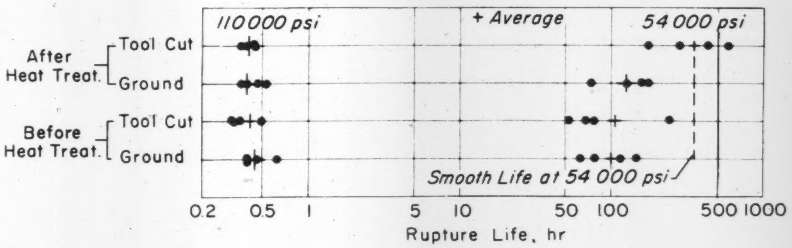

FIG. 7.—1200 F, 110,000 and 54,000 psi. Rupture Life of A-286 Specimens, Notched Before or After Heat Treatment (0.001 in. notch radius).

specimens having 0.001-in. radius notches, tool cut after heat treatment.

In the case of either material, generally, four tests were made at each stress level and for each condition of notch preparation, sharpness, or eccentricity investigated.

## TEST RESULTS

The results obtained in the investigation of notch preparation are shown in Figs. 6 and 7 and those for the influence of eccentricity in Fig. 8. These representations show the effect of the primary variables on the notch-rupture life.

### *Notch Preparation:*

The data for 17-22A(S), Fig. 6, indicate that the 1000 F notch-rupture life of this alloy is essentially identical for notches tool cut or ground before or after

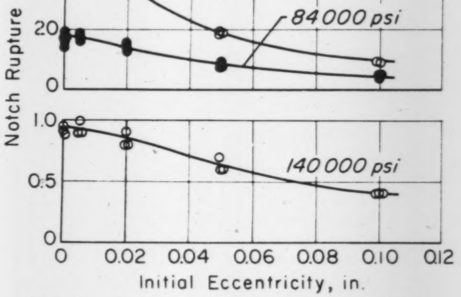

FIG. 8.—Influence of Initial Eccentricity on 1000 F Noch-Rupture Life of 17-22A(S) at Several Stress Levels.

heat treating. This is observed for both 0.001- and 0.0085-in. radius notches tested under conditions yielding either notch-ductile (140,000 psi) or notch-brittle behavior (60,000 psi).

The 1200 F data for A-286, Fig. 7, scatter considerably.[6] However, sufficient data are available to establish an average life and these values are indicated on Fig. 7. At 110,000 psi (notch-rupture strength ratio = 1.15) the rupture life is essentially identical for all conditions of notch preparation investigated. In contrast, tests at the lower stress level (54,000 psi) indicate the longest average life (350 hr) is obtained with notches tool cut after heat treatment. Notches ground after heat treatment have approximately one third this life. Notches tool cut or ground before heat treatment have average lives essentially equal to those ground after heat treatment.

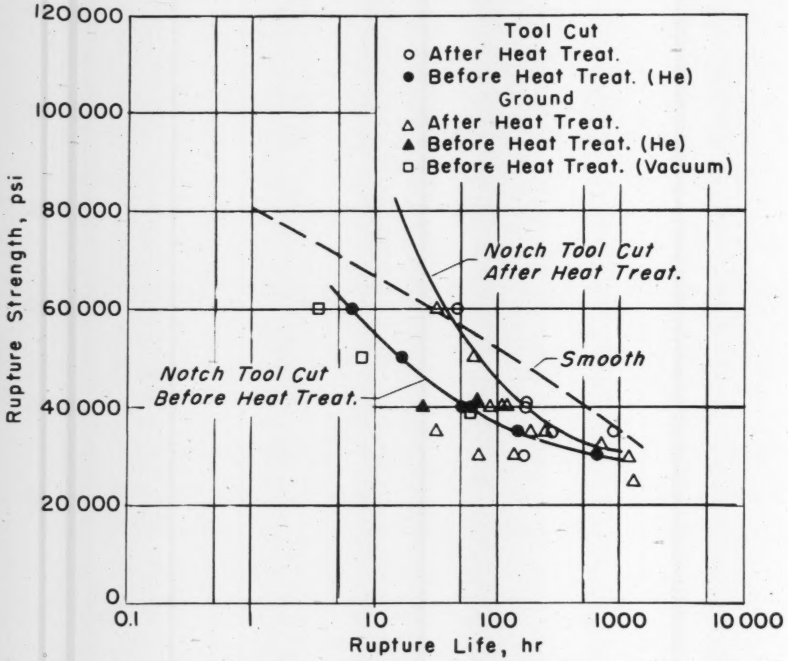

FIG. 9.—Influence of Notch-Preparation Method on the 1350 F Rupture Strength of Inconel X 550 According to Voorhees and Freeman (**7, 8**).

*Eccentricity:*

According to Fig. 8 the notch-rupture life of 17-22A(S) was decreased by an increase in initial eccentricity at all three stress levels investigated. The greatest weakening effect of eccentricity is observed at the stress level (60,000 psi) yielding a notch-brittle condition. At this stress level the notch life continuously decreases with increasing initial eccentricity. The rate of decrease is very rapid at low values of eccentricity and becomes progressively slower as the eccentricity increases. At the two higher stress levels, yielding relatively notch-ductile behavior, the introduction of a small initial eccentricity (up to approximately 0.01 in.) has a negligible effect on the rupture life. Further increases in eccentricity continuously reduce the rupture life at a rate which appears to decrease as the eccentricity increases. The small scatter observed at low values of eccentricity indicates the loading fixtures themselves introduce very little misalignment.

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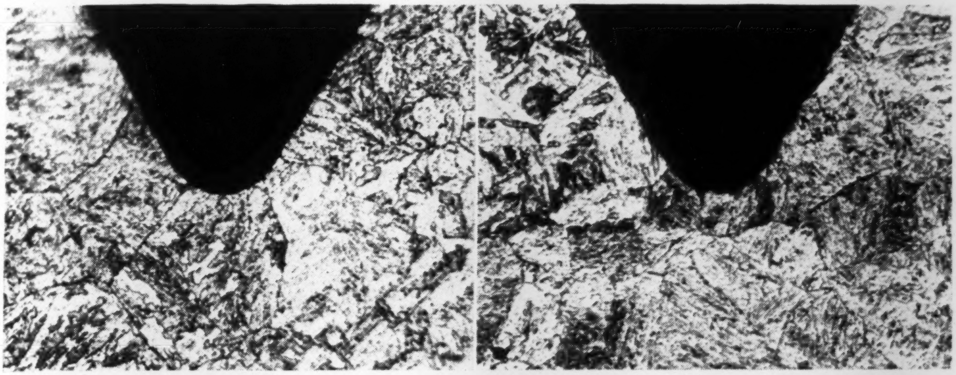
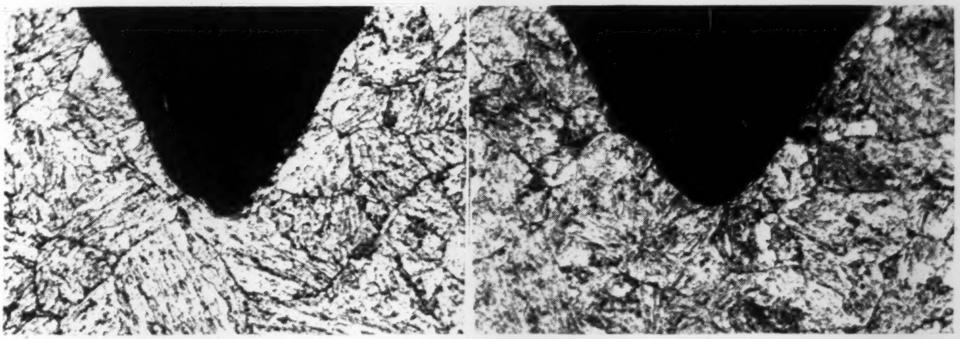
[6] This scatter is believed due primarily to inherent variations in the properties of this complex alloy and not to the testing techniques.



(*a*) Tool cut before heat treatment.

(*c*) Ground before heat treatment.



(*b*) Tool cut after heat treatment.

(*d*) Ground after heat treatment.

FIG. 10.—Photomicrographs of 17-22A(S), 0.001 in. Radius, Notches Tool Cut on Ground Before or After Heat Treatment. Etched in Villella's Martensitic (× 750).

## DISCUSSION OF RESULTS

The data presented in the previous section will be compared with data reported by other investigators and discussed in the light of metallographic examination.

*Notch Preparation:*

*A-286 versus Inconel X-550.*—The results obtained for A-286 tested at 54,000 psi are in many respects in agreement with those for Inconel X-550 tested at 1350 F reported by Voorhees and Freeman (**7, 8**). These investigators employed a notch having a sharpness of approximately 42.5 (0.005-in. radius). Notches were either tool cut or ground before or after a complete heat treatment.[7] For specimens notched before heat treatment, solution was carried out in a raw helium atmosphere and aging was done in air. In addition several specimens machined in the as-received condition were solution treated in vacuum.

The Inconel X-550 results are summarized in Fig. 9. Interpretation of the data at stresses below about 40,000 psi is complicated by severe scatter. For the purposes of this discussion only data above this stress level will be considered. The longest lives are obtained for notches tool cut after heat treatment. Notches ground after heat treatment had only slightly shorter life. Complete heat treatment following tool cutting or grinding results in greatly reduced rupture strength. The authors point out that notches machined before heat treatment in raw helium show distinct evidence of recrystallization, precipitation, and grain boundary oxide penetration. Notches ground before heat treatment did not exhibit these structural alterations when the solution was done in vacuum. However, the notch strength of these specimens was even slightly lower than those solutions treated in raw helium.

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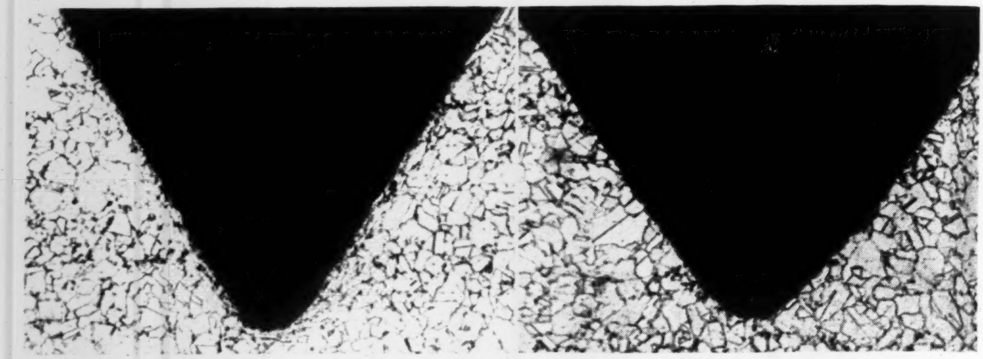
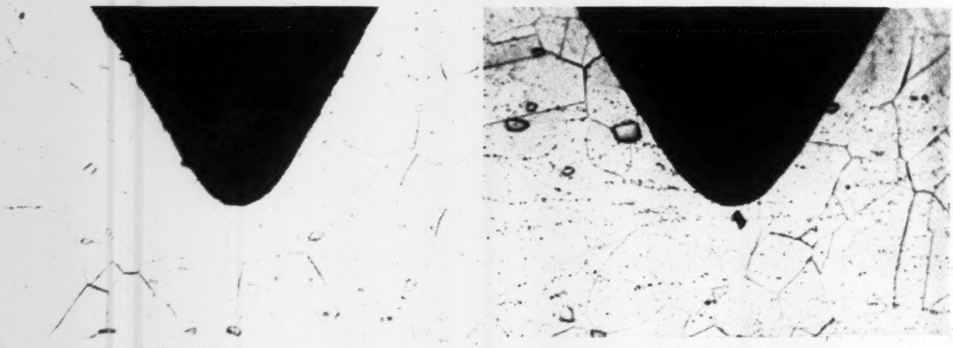
[7] 2150 F, 1 hr air; 1600 F, 4 hr air; 1350 F, 4 hr air.



(*a*) Tool cut as-received. (*b*) Ground as-received.

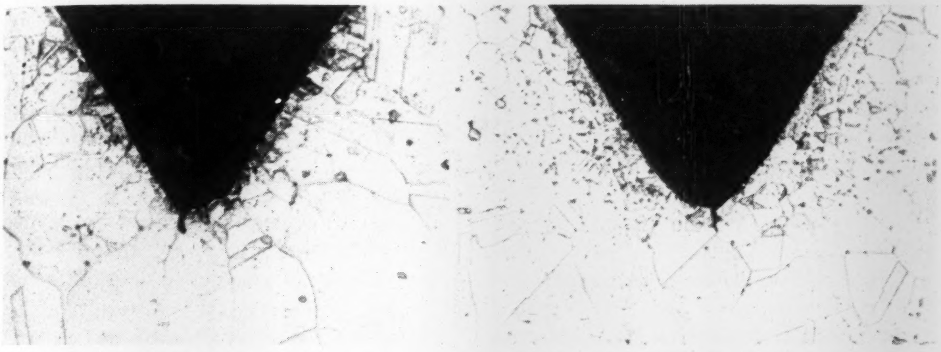


(*c*) Tool cut after heat treatment. (*d*) Ground after heat treatment.

FIG. 11.—Photomicrographs of A-286, 0.001 in. Radius, Notches Machined in the As-Received and in the Heat-Treated Alloy. Electrolytic Etch, 50 per cent $H_2O$, 40 per cent HCl, 10 per cent $HNO_3$ (× 750).

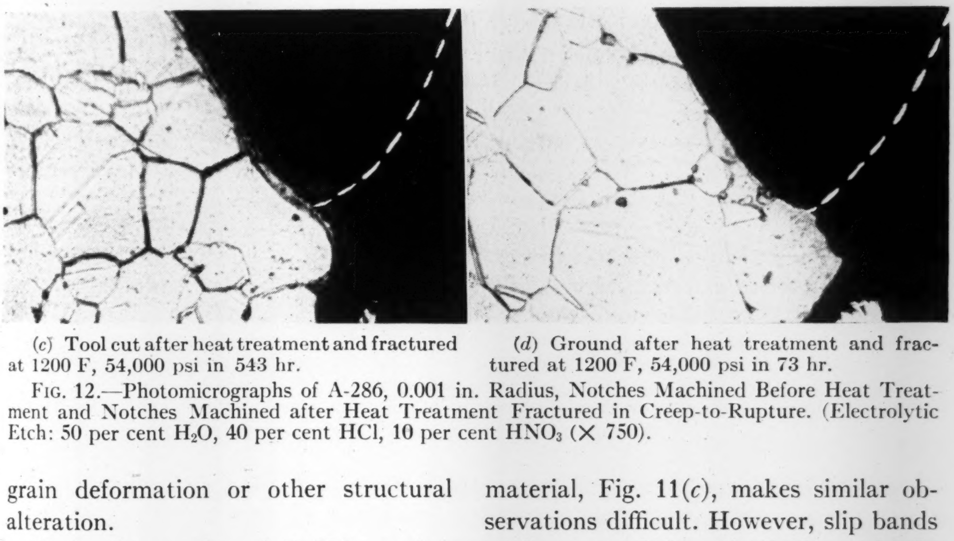
The major difference between the results obtained for Inconel X-550 and those reported here for A-286 is in the relative behavior of notches ground after heat treatment. According to the present results this method of notch preparation yields considerably lower rupture life than tool cutting. In contrast, the results for Inconel X-550 show little difference between notches tool cut and ground after heat treatment.

*Metallographic Examination.*—Photomicrographs of 0.001-in. radius notches[8] of 17-22A(S) are shown in Fig. 10. Tool cutting or grinding either before or after heat treating yields essentially identical structures which show no evidence of grain deformation or other structural alteration.



(*a*) Tool cut before heat treatment.

(*b*) Ground before heat treatment.



(*c*) Tool cut after heat treatment and fractured at 1200 F, 54,000 psi in 543 hr.

(*d*) Ground after heat treatment and fractured at 1200 F, 54,000 psi in 73 hr.

FIG. 12.—Photomicrographs of A-286, 0.001 in. Radius, Notches Machined Before Heat Treatment and Notches Machined after Heat Treatment Fractured in Creep-to-Rupture. (Electrolytic Etch: 50 per cent $H_2O$, 40 per cent HCl, 10 per cent $HNO_3$ (× 750).

Photomicrographs of A-286 notches are shown in Figs. 11 and 12 which illustrate the structure at the notch root for tool cut and ground notches. Tool cutting the as-received material, Fig. 11(*a*), apparently produces severe compressive deformation (burnishing) in a narrow band (approximately 0.0003 in. deep) along the notch flank and at the root. The larger grain size of the heat-treated material, Fig. 11(*c*), makes similar observations difficult. However, slip bands at the root and along the flank are clearly evident in visual examination. In contrast, grinding either the as-received, Fig. 11(*b*), or the heat-treated, Fig. 11(*d*), alloy produces no evidence of deformation at ×750.

Specimens of A-286 notched before

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[8] There is no detectable difference in microstructure between 0.001-in. and 0.0085-in. radius notches.

heat treatment, Figs. 12(*a*) and (*b*),[9] show recrystallization, precipitation, and possibly some grain oxide penetration. The maximum depth of the disturbed layer was about 0.0015 in. These alterations are similar to those reported by Voorhees and Freeman for Inconel X-550. They attribute them to the heat treating technique and point out no such structural changes were observed when heat treatment was done in vacuum. The authors agree with this explanation.

Specimens notched after heat treatment and tested in creep rupture at 1200 F, are shown in Figs. 12(*c*) and (*d*). The only change from the as-machined condition, Figs. 11(*c*) and (*d*), was grain boundary widening in the highly deformed zone at the notch root. This, as might be expected, is more pronounced for the specimen which had longest life.

*Explanation of Results.*—Voorhees and Freeman (**7**) have suggested that the influence of method of notch preparation on the rupture strength of Inconel X-550 can be related to residual stresses produced by machining. The present results are consistent with such an explanation.

There is considerable literature relating to the residual stresses produced by grinding (**11, 12, 13, 14, 15**) and some information regarding those produced by tool cutting (**16**). These investigations are concerned with machining of plane surfaces so that deflection measurements of the workpiece may be used in the stress calculation. According to Letner and his co-workers (**11, 12, 13**) grinding results in a biaxial stress state. The stress distribution fluctuates near the surface. Thus, if biaxial tension exists at the surface a region of biaxial compression is found a few ten thousandths of an inch below the surface. The sign of the surface stresses, their magnitude, and the depth of the stressed region varies with both the machining variables and the metal structure. According to Henriksen (**16**) similar complexities may be expected for tool cutting with the exception that only surface tensile stresses are reported. Tarsov (**15**) has emphasized that very gentle grinding conditions involving relatively soft wheels, proper lubricants, and downfeeds less than 0.0005 in. produce very low residual stresses. It is not possible to translate the results obtained for plane surface machining directly to notching. However, they may yield a qualitative indication of the relative severity of the machining methods employed here. Thus, the present notch grinding conditions (see Appendix) are considered gentle and would be expected to produce very low residual stresses. The information available on residual stresses produced by tool cutting is probably not applicable here since, as mentioned previously, there is evidence of a very thin burnished (compressed) layer on the flanks and at the notch root. In all cases it would seem reasonable to assume that heat treatment after notching completely removes any residual stresses.

With these considerations in mind it is possible to arrive at a tentative explanation of the results. The fact that specimens of 17-22A(S) notched before or after heat treatment have essentially identical rupture lives and identical and undisturbed microstructures indicates the machining methods employed result in negligible residual stresses for this alloy. It might be argued that residual stresses would be relieved during testing at 1000 F. However, stress relaxation tests (**17**) on conventionally heat-treated 17-22A(S) indicate that not more than about half of any residual stress would be removed in 1000 hr at 1000 F. The heat treatment used in this investigation should result in even lower relaxation rates.

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[9] A certain amount of the dark coloration in these photomicrographs is staining which is very difficult to avoid due to the presence of slight cracks between the bakelite and the metal.

In contrast to 17-22A(S), the rupture life of A-286 was distinctly influenced by the method of notch preparation when the test conditions yielded low notch ductility. It should be remembered that the relatively high strain hardening capacity of this alloy renders machining much more difficult than for 17-22A(S). The longest lives were obtained for A-286 notches tool cut after heat treatment and this may be explained by the presence of compressive stresses in the burnished layer (see Fig. 11(*a*)). Notches ground after heat treatment show no evidence of such surface deformation (see Fig. 11(*d*)) and have lives which are essentially identical to specimens either tool cut or ground before heat treatment. It is therefore thought that the careful notch grinding used in this investigation produced negligible residual stresses in this alloy.

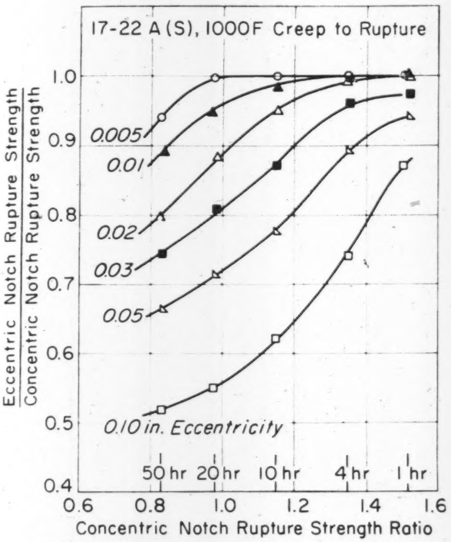

FIG. 13.—Ratio between Eccentric and Concentric Notch-Rupture Strength as a Function of the Concentric Notch-Rupture Strength Ratio for Various Initial Eccentricities.

Identical life was obtained for all conditions of notch preparation when A-286 specimens were tested under conditions yielding relatively high notch ductility. This may be explained by considering the relatively high plastic flow associated with testing at the higher stress. This deformation wipes out the residual stress.

A-286 Specimens heat treated after machining show evidence of considerable structural alteration (see Figs. 12(*a*) and (*b*)). As mentioned previously, this is believed to occur during the heat treatment. However, its presence is not thought to contribute significantly to the reduction in life observed at the low stress inasmuch as notches ground after heat treatment are free of these disturbances and have essentially identical properties. In addition, the rupture life of such specimens was identical to those machined after heat treatment when tested at the higher stress. Furthermore, Voorhees and Freeman report that vacuum heat treatment after notching produced clean structures in Inconel X-550 with slightly lower strengths (see Fig. 9) than were obtained using a helium atmosphere which did result in structural alteration.

*Eccentricity:*

It might be expected that the effect of initial eccentricity would vary depending on the capacity of the notch to deform under the test load and temperature. As shown previously (**5**, **18**) the notch-rupture strength ratio is an indicator of this deformation capacity.

The primary eccentricity data in Fig. 8 may be replotted as a function of the concentric notch-rupture strength ratio. To develop this plot the notch-rupture strength isothermal, Fig. 4, was taken as a guide in establishing the trend of the notch-rupture strength isothermals at the various levels of eccentricity investigated. The smooth rupture-strength isothermal from Fig. 4 was used in calculating the concentric notch-rupture strength ratio. At a given value of initial eccentricity the ratio between the eccentric and the concentric notch-rupture strength has been used as a measure of the influence of eccentricity. This ratio determined at several different rupture times between 1 and 50 hr is plotted against the corresponding concentric notch-rupture strength ratio in Fig. 13. A similar plot for the room temperature data obtained by Sachs, Lubahn, and Ebert (**5**) is shown in Fig. 14. Comparing Figs. 13 and 14, certain similarities are noted between behaviors in room temperature tension tests and creep-to-rupture tests. The weakening effect of eccentricity increases with decreasing concentric notch strength ratio and this decrease becomes more pronounced as the eccentricity increases. Distinct dissimilarities are also noted. In creep-to-rupture tests the smallest eccentricities produce definite weakening at low values of the concentric notch-rupture strength ratio. In the tension test considerably larger eccentricities are necessary to produce similar effects.[10] The effect of eccentricity in tension tests develops rapidly with small decreases in the concentric notch strength ratio from its maximum value (approximately 1.5). On the other hand, in the creep-to-rupture test the weakening effect of eccentricity develops gradually and low eccentricities (<0.01 in.) have practically no effect if the concentric notch-rupture strength ratio is greater than unity.

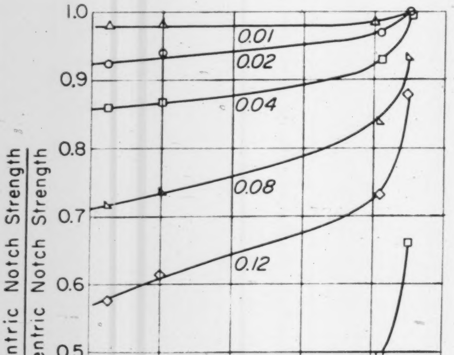

FIG. 14.—Ratio between Eccentric and Concentric Notch Strength as a Function of Concentric Notch Strength Ratio for Various Initial Eccentricities (**5**).

The influence of initial eccentricity and its variation with the concentric notch strength ratio may be explained as follows. In the elastic state the eccentricity results in a bending stress being superimposed on the high surface stress associated with the sharp notch. Deformation of the specimen results in the specimen axis approaching the loading axis with consequent reduction in bending stress. Thus, the effect of a given eccentricity would be expected to increase with a decrease in the concentric notch strength ratio (notch ductility). Deformation of the specimen also produces a strain gradient across the notch section which replaces the stress gradient associated with the initial eccentricity. This strain gradient if large can reduce the average strength of the material. Thus, the most highly deformed portion of the notch reaches its fracture strain at a relatively low average stress in the tension test and at a relatively low time in the creep-to-rupture test. Therefore, high eccentricities would be expected to have a weakening effect even on materials with high concentric notch strength ratios.

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[10] The specimens employed by Sachs, Lubahn and Ebert (**5**) were essentially identical to those used in the present investigation with the exception that the notch diameter was approximately 17 per cent larger. Assuming constant moment bending of a cylindrical bar, a 17 per cent higher eccentricity would be needed to produce the same bending stress in the larger specimen. This dimensional effect is felt to be too small to account for the greater effect of eccentricity observed in creep-to-rupture tests.

## PRACTICAL SIGNIFICANCE OF THE RESULTS

The notch preparation method can have either practically negligible or large influence on the rupture strength. The magnitude of this effect has been shown to be dependent on the alloy and its seems reasonable to assume that it also will be a function of the heat treatment and testing conditions.

The behavior observed in this investigation for the chromium-molybdenum-vanadium steel 17-22A(S) is probably typical for most modified low alloy and ferritic chromium steels. This may be inferred from the fact that these materials are generally heat treated to the same range of hardness (Rockwell hardness C-30 to 35) for elevated temperature service. Furthermore, their machining characteristics are very similar. Thus, for these alloys the method of notch preparation should have negligible effect on the rupture strength provided gentle methods of finish maching are used. However, lower hardness levels may behave somewhat differently and it has been shown (12) that grinding stresses in steel generally increase with decreasing hardness.

On the other hand, results obtained for A-286 in this investigation and for Inconel X-550 by Voorhees and Freeman (7, 8) indicate the rupture strength of austenitic heat resisting alloys is very likely to be influenced by the machining method. It is exceedingly difficult to tool cut sharp notches into materials having high strain hardening rates without burnishing the root and flanks of the notch. Such materials are also very sensitive to the grinding conditions. However, with very gentle grinding it appears possible to greatly minimize residual stresses. Other investigators have pointed out that the machining method can in some cases influence the alloy structure developed on subsequent heating near the aging temperature. These effects may also influence the rupture strength. The above mentioned factors unfortunately complicate interpretation of notch rupture data for design purposes. Notch specimens of austenitic alloys should be machined in a condition of heat treatment and in a manner as close as possible to that used for the finished part.

Regarding the influence of eccentricity, the present results show conclusively that small misalignments between the load axis and longitudinal specimen axis can reduce the notch-rupture strength if the concentric notch-rupture strength ratio (notch ductility) is low. Under such conditions, the influence of misalignment in rupture tests can be even greater than that observed in tension tests of hardened steels. Apparently a significant portion of the scatter frequently observed in notch-rupture data is due to the misalignment introduced by conventional threaded loading devices. As has been previously shown (9) these misalignments vary widely from test to test in a random manner. The result, of course, is a variation in the notch strength of identical specimens presumably tested under the same conditions. The small scatter observed in notch data for 17-22A(S) (see Figs. 6 and 8) is a good indication that the loading devices employed in the present investigation introduce very little eccentricity. When tension testing notch specimens of high strength level, heat-treated steels at room temperature, it is generally recognized that to obtain reproducible data special precautions must be taken to reduce misalignments. It is certainly to be recommended that similar precautions be taken in notch-rupture tests.

The results on influence of eccentricity have further implications. Thus, a material which does not show notch weakening in the laboratory creep test may be notch weakened in service if the component contains stress concentrations and is subjected to bending loads. This is not an infrequent occurrence and when such service conditions are present a notch test incorporating a controlled amount of eccentricity should be considered.

*Acknowledgments:*

The authors are indebted to Messrs. J. W. Freeman and H. R. Voorhees of the University of Michigan for permission to include their data on Inconel X-550 in this paper. Also appreciation is expressed to Mr. T. H. Guzik, Head of Special Projections Section, Fabrication Division, Nat. Advisory Committee for Aeronautics, Lewis Laboratory, who supervised the development of the grinding techniques used in notching the specimens.

## APPENDIX

## NOTCH MACHINING TECHNIQUES

*Tool Cutting:*

The tool cut notches employed in this and previous Nat. Advisory Committee for Aeronautics (NACA) investigations were produced by the Walters Co.[11] Exact details of the machining techniques are proprietary. However, certain steps in the procedure can be stated. Notches in 17-22A(S) were cut with Kennametal K-6 tools while Carboloy 883 was used for the A-286. If a radius greater than 0.001 in. was desired, the tool was diamond ground to contour using a special fixture. In all cases the notch was roughed to within 0.010 to 0.015 in. of final diameter. For finishing, the tool was ground on a 400 grit diamond wheel. Finishing cuts were in the order of 0.0002 to 0.0003 in. per revolution using a head stock speed of about 45 rpm. Lubricant for the 17-22A(S) was a water soluble oil and for the A-286 a paste compound called Anchorlube.[12]

*Grinding:*

Specimens were notched at NACA on an "Excello" thread grinder using a 320-J resinoid, 18-in. diameter wheel and "top" thread grinding oil.[13] The wheel speed was 2425 rpm, yielding a surface speed of approximately 11,500 ft per min. Stock speed was approximately 4 rpm.

Sharp notches were ground to about 0.002 in. oversize with hand feed of approximately 0.001 in. per revolution of the stock. The wheel was then dressed to "zero" radius and the notch finished using a hand feed of approximately 0.00005 in. per revolution. The 0.0085-in. radius notches were ground with a sharp contour wheel to within the 0.002 in. final diameter. The wheel was then hand dressed to final contour and the notch finished using a hand feed of 0.00005 in. per revolution of the stock. Generally, about eight specimens could be finish-ground before the notch radius exceeded 0.009 in.

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[11] 876 E. 200 St., Cleveland 19, Ohio.

[12] Anchor Chemical Co., 10721 Bridge Ave., Cleveland, Ohio.

[13] Rex Oil and Chemical Co., 9120 Loren Ave., Cleveland, Ohio.

## REFERENCES

(1) R. L. Carlson, R. J. MacDonald, and W. F. Simmons, "Factors Influencing the Notch Rupture Strength of Heat Resistant Alloys at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 78, Feb., 1956, pp. 349–358.

(2) F. H. Vitovec and B. J. Lazan, "Creep Rupture and Notch Sensitivity Properties of S-816 Alloy up to 1650 F Under Fatigue and Static Stress," Symposium on Metallic Materials for Service at Temperatures Above 1600 F, pp. 69–89, Am. Soc. Testing Mats., (1955). (Issued as separate publication *ASTM STP No. 174.*)

(3) F. H. Vitovec and B. J. Lazan, "Fatigue, Creep and Rupture Properties of Heat Resistant Material," *Technical Report No. 56-181*, Wright Air Development Center, Aug., 1956 (ASTIA No. AD 97240).

(4) W. S. Hyler and W. F. Simmons, "Factors Influencing the Notch Fatigue Strengthening of N-155 Alloy at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 78, pp. 339–348 (1956).

(5) G. Sachs, J. D. Lubahn, and L. J. Ebert, "Notch Bar Tensile Test Characteristics of Heat Treated Low Alloy Steels," *Transactions*, Am. Soc. Metals, Vol. 33, pp. 340–395 (1944).

(6) E. A. Davis and M. J. Manjoine, "Effect of Notch Geometry on Rupture Strength at Elevated Temperatures," Symposium on Strength and Ductility of Metals at Elevated Temperatures, pp. 67–88, Am. Soc. Testing Mats. (1952). (Issued as separate publication *ASTM STP No. 128.*)

(7) H. R. Voorhees and J. W. Freeman, "Notch Sensitivity of Heat Resistant Alloys at Elevated Temperatures," *Technical Report No. 54-175*, Part 2, Wright Air Development Center, Dec., 1954.

(8) H. R. Voorhees and J. W. Freeman, "Notch Sensitivity of Heat Resistant Alloys at Elevated Temperatures," *Technical Report No. 54-175*, Part 3, Wright Air Development Center, July, 1956.

(9) M. H. Jones and W. F. Brown, Jr., "An Axial Loading Creep Machine," ASTM BULLETIN, No. 211, Jan. 1956, pp. 53–60 (TP 25).

(10) M. H. Jones, D. P. Newman, G. Sachs, and W. F. Brown, Jr., "Investigation of Variation in Normalizing and Tempering Procedure on Stress Rupture Strength, Creep Embrittlement and Notch Sensitivity for a Chromium-Molybdenum-Vanadium and 17 Chromium-4 Nickel-4 Copper Steel," *Transactions*, Am. Soc. Metals, Vol. 47, pp. 926–957 (1955).

(11) H. R. Letner and J. H. Snyder, "Grinding and Lapping Stresses in Manganese Oil Hardening Tool Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 75, pp. 873–882 (1953).

(12) H. R. Letner, "Residual Grinding Stresses in Hardened Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 77, pp. 1089–1098 (1955).

(13) H. R. Letner, "Influence of Grinding Fluids Upon Residual Stresses in Hardened Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 79, Jan., 1957, pp. 149–153.

(14) L. V. Colwell, M. J. Sinnott, and J. C. Tobin, "The Determination of Residual Stresses in Hardened Ground Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 77, pp. 1099–1105 (1955).

(15) L. P. Tarsov, Discussion of Frisch and Thomsen paper, *Transactions*, Vol. 73, pp. 344–345 (1951).

(16) E. K. Henriksen, "Residual Stresses in Machined Surfaces," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 73, pp. 69–76 (1951).

(17) Resume of High Temperature Investigations Conducted during 1955–1956, Timken Roller Bearing Co., Steel and Tube Division, Canton, Ohio.

(18) D. P. Newman, M. H. Jones, and W. F. Brown, Jr., "Time-Temperature Dependence of the Notch Effect and Influence of Notch Depth in Stress Rupture Tests on a Chromium-Molybdenum-Vanadium Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, pp. 677–692 (1953).

(19) W. F. Brown, Jr., M. H. Jones, and D. P. Newman, "Influence of Sharp Notches on the Stress-Rupture Characteristics of Several Heat-Resisting Alloys," Symposium on Strength and Ductility of Metals at Elevated Temperatures, p. 25, Am. Soc. Testing Mats. (1952). (Issued as separate publication *ASTM STP No. 128.*)

(20) W. F. Brown, Jr., M. H. Jones, and D. P. Newman, "Influence of Sharp Notches on the Stress-Rupture Characteristics of Several Heat-Resisting Alloys, Part II," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, pp. 661–676 (1953).

# DISCUSSION

MR. L. P. TARASOV[1] (*presented in written form*).—The authors have shown that careful notch grinding in heat-treated A-286 had no effect on its rupture life under low notch-ductility test conditions and they tentatively attribute this to the absence of appreciable residual grinding stresses. This interpretation, while fully justified on the basis of the published information available to the authors, needs to be reviewed in the light of the most recent studies of grinding stresses and their effects.

Although the authors mention the writer as stating that very gentle grinding conditions produce very low residual stresses, he actually referred to low deflections and not to low stresses. The reason for making this distinction is that the deflection of a thin strip will be small when both tensile and compressive stresses are present if the resultant bending moments happen to be about equal in magnitude or if the stressed layer is extremely shallow. Thus negligible deflections do not necessarily mean that the residual stresses are equally negligible.

In some very gently ground specimens of hardened steel, for which the unit downfeed was only 0.0001 in., Letner[2, 3] has found tensile stresses alone, in others both tensile and compressive stresses. (Only stresses parallel to the grinding direction are considered here.) Their magnitude was of the order of 20,000 or 30,000 psi, although considerably higher or lower values were observed in individual specimens. Thus in hardened steel, at least, the residual stresses obtained under very gently grinding conditions (very small downfeed) are not particularly low, and they can fluctuate unpredictably from one specimen to another.

The only grinding stress data for high-temperature alloys are those that have just been obtained by Halverstadt[4] who found that a residual compressive stress of about 20,000 psi was developed in A-286 when this was ground with oil, the unit downfeed being 0.0005 in. Similar results were obtained with two other alloys.

Residual stresses of this magnitude can hardly be considered negligible, but they are not high in relationship to the yield stress. Perhaps the best characterization would be low or moderate. The important thing about these stresses is not so much their magnitude as the effect that they may have upon various mechanical properties. It has been found[3] that even a residual tensile stress of 50,000 psi in hardened steel, obtained by grinding under good commercial conditions, had no measurable effect upon the reversed bending fatigue limit Thus the fact that the authors observed their notch-grinding procedure to have no effect upon the rupture life does not necessarily imply the absence of appreciable residual stresses. Only an actual stress determination would show whether or not the stress was really negligible.

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[1] Research and Development Department, Norton Co., Worcester, Mass.

[2] H. R. Letner, "Residual Grinding Stresses in Hardened Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 77, pp. 1089–1098 (1955).

[3] L. P. Tarasov, W. S. Hyler, and H. R. Letner, "Effect of Grinding Conditions and Resultant Residual Stresses on the Fatigue Strength of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 601 (1957).

[4] R. D. Halverstadt, "Analysis of Residual Stress in Ground Surfaces of High-Temperature Alloys," *Preprint No. 57-SA-62*, Am. Soc. Mechanical Engrs., June, 1957.

In the fatigue study just cited, it was found that certain grinding conditions caused an appreciable increase of the fatigue limit of hardened steel above its par value. This strengthening was attributed at least in part to the beneficial effect of cold work separate from the effect of residual stress since the fatigue limit did not increase under another set of grinding conditions that produced essentially the same residual stress pattern. The authors have shown that cutting the notches in heat-treated A-286 improved the rupture life considerably, an effect which they ascribe to the burnishing action of the carbide tool. It is interesting to speculate that a similar effect might have occurred had the notches been ground at a considerably higher infeed rate.

From the standpoint of evaluating the notch-rupture life of a material, such strengthening effects should naturally be avoided. However, these same effects may be of considerable practical value in increasing the rupture life of actual parts under certain conditions. Thus it would seem desirable to investigate a variety of grinding and cutting conditions to see whether these operations can be used not only to impart the desired form to the finished product but also to strengthen it.

MESSRS. H. R. VOORHEES[5] AND J. W. FREEMAN[5] (*by letter*).—The authors have shown that rupture life of notched specimens may or may not be influenced to a marked extent by methods of notch preparation or by eccentricity of loading during testing, depending on the material and test conditions. This result might be expected because the same factors should be involved as govern the influence of the notch itself. The only new factor is possible alteration of the properties of the material in the critical section at the base of the notch. The notch produces a concentrated complex stress. Notch preparation may add residual stresses superimposed on the geometrical stress effects and also may disturb the metal near the surface. Eccentricity simply modifies the stresses at the notch.

It follows then, as the paper demonstrates, that when the properties of the alloy and test conditions permit adjustment to the stress concentration (notch strengthening) the preparation and eccentricity factors have little influence. When they do not (notch weakening) then the superimposed effects can have a pronounced effect, as was observed. Erratic test results are to be expected when preparation and eccentricity vary between specimens of material prone to notch weakening. It is equally true that if residual stresses or eccentric loading were uniformly present in all specimens, consistent but erroneous results could be obtained.

Other sources of erratic results should be recognized. Yielding at the notch root during initial loading may be a very important factor in subsequent life of a notched specimen. If such yielding is borderline in setting up conditions for notch weakening or notch strengthening, then small variations in yield strength between specimens could be an important source of variability in the notch tests.

Extreme caution should be exercised in applying the conclusions suggested by both the tests of the present paper and results of reference (**8**) of the paper, namely that favorable residual stresses introduced by a tool cut after heat treatment will produce the longest rupture life in alloys subject to work hardening during machining. Indeed, for certain alloys a relatively small amount of plastic deformation can result in marked deterioration of subsequent rupture strength.

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[5] Research Associate, Engineering Research Institute, University of Michigan, Ann Arbor, Mich.

In summary, to evaluate testing methods adequately for notched bars and to interpret the results, one needs to know what factors (yield properties, work-hardening characteristics, creep and rupture properties, susceptibility to strain damage) control behavior for the particular material and procedures used. To apply notched-bar data freely without such knowledge may lead to completely false conclusions.

MESSRS. J. L. SHANNON, JR., M. H. JONES AND W. F. BROWN, JR. (*authors' closure*).—The authors wish to thank Messrs Tarasov, Voorhees and Freeman for their interesting comments.

Tarasov's statement that low deflections of the work piece are not necessarily an indication of low residual stresses is correct, since the stresses may be high but vary in sign near the surface. His method of residual stress indication may be used only with thin, flat strips, and the results therefrom are not necessarily applicable to ground notches. However, it appears reasonable to assume that those grinding conditions which would minimize the magnitude of the residual tensile stresses and the depth of the affected volume in a flat, thin strip would also minimize the residual stresses in a notched work piece.

Letner's work (**11, 12, 13**)[6] appears to generally support the assumption that, other things being constant, reducing the downfeed would tend to reduce the magnitude and depth of penetration of stresses. The authors therefore believe their gentle infeed rate of 0.00005 in. per rev (still milder than Letner's most gentle condition) produces relatively low residual stresses. In any event, the data indicate whatever stresses may be present do not significantly affect the measured mechanical properties.

The results of Halverstadt[4] also appear to support the above assumption. Thus, A-286 ground under gentle conditions (0.0005 in. downfeed) had stresses less than 20,000 psi and a stress penetration depth of less than 0.006 in. Further results given for M-252 indicate it is possible to grind with no measurable residual stress. It also should be remembered that Halverstadt considers his probable error as about ±10,000 psi with higher errors at the surface.

Tarasov indicates that residual tensile stresses as high as 50,000 psi had no effect on SAE 52100. We have examined his very interesting paper and are certainly surprised at the results. It is rather difficult to comment on the conclusions since $S$-$N$ curves for the various groups were not published. In this connection we feel that a large number of specimens is necessary to establish the $S$-$N$ curve, particularly in the region of a "fatigue limit." Sachs[7] has recently made an investigation of the rotating-beam fatigue strength of SAE 52100 (50 to 65 Rockwell hardness, C scale). From his results it may be concluded: (1) the cycles to failure at a given stress level can vary by more than one order of magnitude. Consequently, 30 or more samples were necessary to establish the "endurance limit" even though the majority of the tests were concentrated at the low stresses. (2) In spite of high retained quenching stresses in samples with hardnesses above Rockwell C 56, the fatigue strength at $10^8$ cycles remains essentially constant from Rockwell C 54 to C 62. (3) The residual stresses do, however, result in a decrease in the proportional limit, yield strength, and tensile strength above a hardness of about Rockwell C 58. The most important conclusion to be drawn from Sachs' results is that the influence of residual stresses on fatigue behavior may not necessarily reflect similar behavior in other mechanical properties.

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[6] See list of references appended to the paper, p. 849.

[7] G. Sachs, A. T. Viggiano and R. J. Sell, "Tension, Compression and Fatigue Tests on Several Tool Steels to Determine Their Suitability as Roller Contact Bearing Materials," *Report No. MET 415-5710T2*, Syracuse University (1957).

The authors have no information concerning the influence of high infeed rates. However, it is possible that local overheating could occur which would increase the magnitude of residual tensile stresses. Generally speaking, the problem of selecting machining procedures to strengthen the material is very difficult to solve. For some materials the presence of cold work may be detrimental. In this connection, Letner[8] has pointed out that the safest procedure is to produce a stress free surface.

Messrs Voorhees and Freeman appear to be in general agreement with the interpretation of the data as presented. The authors agree with all the points made in their discussion and wish to further emphasize the danger of generalizing on the beneficial effects of tool cutting.

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[8] H. R. Letner, A. B. Sauvageot, "Relief of Residual Grinding Stresses by Annealing," *Metal Progress,* Sept., 1957, pp. 79–82.

# CONSTANT-STRESS CREEP RUPTURE TESTS OF A KILLED CARBON STEEL*

By P. N. Randall[1]

## Synopsis

The unique features of the test equipment described are: means for maintaining constant stress throughout the test by a reduction of the lever ratio as the specimen stretches; provision for adjustment of axiality of load, continuous recording of elongation; and temperature control by a servo system which adjusts voltage to the furnace continuously. The paper presents stress rupture data and also true strain rate *versus* time data for 40 pairs of specimens tested to rupture at five temperatures from 800 to 1200 F and for times ranging from 0.05 to 15000 hr. Two minima in the creep rate were found in the longer tests at the three lower temperatures. Tests to find an explanation are described.

The choice of constant-stress creep rupture equipment was a result of beliefs that useful information would be wasted if creep rates were not measured throughout the test, and that maintaining the stress constant would simplify the interpretation of the creep rate data. This is hardly an original thought, for the pioneering work in creep by Andrade (**1**)[2] was done on constant-stress equipment, one feature of which was copied in the present method.

The material chosen for the investigation was a carbon steel (A201) widely used for pressure vessels in the petroleum industry. Five temperatures were used, ranging from 800 to 1200 F, and stresses were chosen which would give a variation in rupture life from about 0.1 hr to about 10,000 hr except at the two higher temperatures.

The paper presents test data on rupture life and ductility and compares these with published data obtained on constant-load equipment for steels of similar composition. The actual departures from constancy of stress in the present tests are described.

The creep rate data presented exhibit an unusual feature; the occurrence of two minima in the creep rate, chiefly for the longer tests at the three lower temperatures. In order to explain this phenomenon, some tests are described in which the specimen received a heat treatment prior to the test.

## Description of Test Equipment

The machines were designed to maintain constant stress in the specimen regardless of the elongation, provided: (*a*) the length of the section which is elongating is equal to the length assumed in the design, (*b*) the reduction of area within this region is uniform, and (*c*) the principle of constant volume applies. To maintain constant stress, the load must decrease as the specimen cross-sectional area decreases. The area is inversely proportional to the elongation of the specimen if provisions (*b*) and (*c*) apply. Elongation in turn controls the movement of the lever, which is so constructed that the lever ratio decreases as the lever rotates about the fulcrum, as shown in Fig. 1. The moving pivot, directly above the furnace, is lower than the fulcrum. Hence it moves outward as well as upward as the specimen stretches, thus causing the lever ratio to decrease. In addition, the perpendicular distance from the fulcrum to the cable which supports the weights decreases in length as the lever rotates. The use of the curved surface on which the cable rests was an idea taken from the work of Andrade (1). A check of the decrease in lever ratio with specimen elongation, by means of a weigh bar bearing SR-4 strain gages, showed the design requirements were fulfilled with a maximum error of 0.8 per cent.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Senior Research Supervisor, Engineering Research Department, Standard Oil Co., (Indiana), Whiting, Ind.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 870.

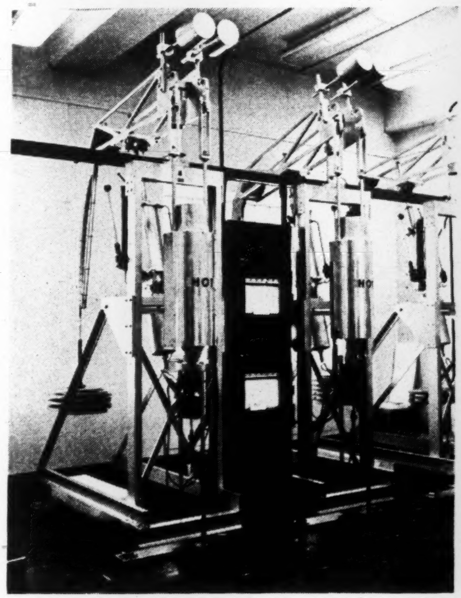

FIG. 1.—General View of Creep-Rupture Machines.

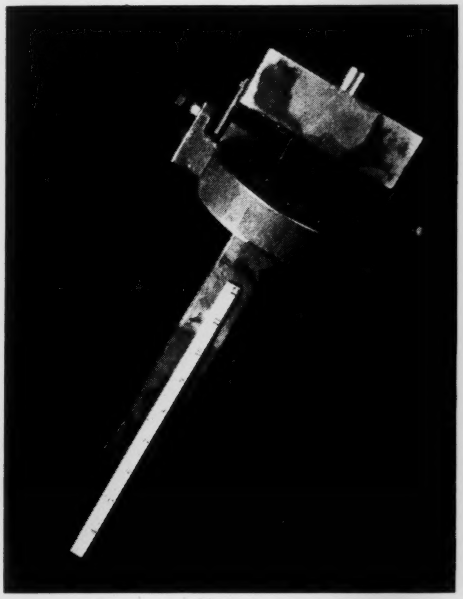

FIG. 2.—Pair of Crossed Knife Edges and Pull Rod.

To start a test, the desired amount of dead weight is placed on the scale pan, supported from the floor, and the load is applied to the specimen by means of the hydraulic jack which supports the fulcrum of the lever system. This requires between 1 and 2 min, hence provides a gradual application of the load to the specimen. The constant stress feature requires that the no-load position of the lever, with the pull rods seated, would be level. This is determined by trial prior to putting weights on the pan.

To provide for axiality, the load is applied through a pair of adjustable crossed knife edges as shown in Figs. 1 and 2. The adjustment is done with the weigh bar substituting for the specimen. Weights are applied and bending stresses are eliminated by adjusting the pair of knife edges on top of the furnace for bending in one plane and the pair below the furnace for bending in the plane at right angles to the first. Bending stresses in the weigh bar are normally reduced to not more than 1 per cent of the average stress. The pull rod seating surfaces are such that it was found possible to remove and replace the weigh bar with little change in the bending stress.

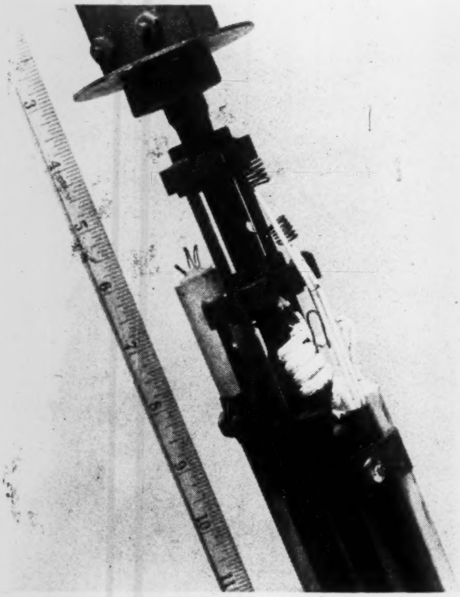

FIG. 3.—Specimen with Thermocouples and Control Thermopile. From an Interrupted test.

The temperature controller is an original design by other members of the author's department. The sensing element is a three-junction platinum-platinum rhodium thermopile, supported alongside but not touching the specimen, as shown in Fig. 3. The output of this thermopile is matched against a reference voltage by a servo system which amplifies the voltage difference and operates a motor-driven autotransformer. Power to the furnace is thereby continuously adjusted to minimize the error voltage.

Two thermocouples are wired to the specimen just below the lower gage point as shown in Fig. 3. A Chromel-Alumel thermocouple, connected to a recorder, is used to detect short-time instabilities or upsets. A platinum-platinum rhodium thermocouple, read twice daily with a precision potentiometer, is used to check long-time stability. The accuracy of control was normally better than ±1 F for the entire test. There were many periods of 10 days duration in which the variation in indicated temperature was less than ± 0.2 F. To determine differences in temperature along the length of the specimen, a differential thermocouple method is used. Three Alumel wires are

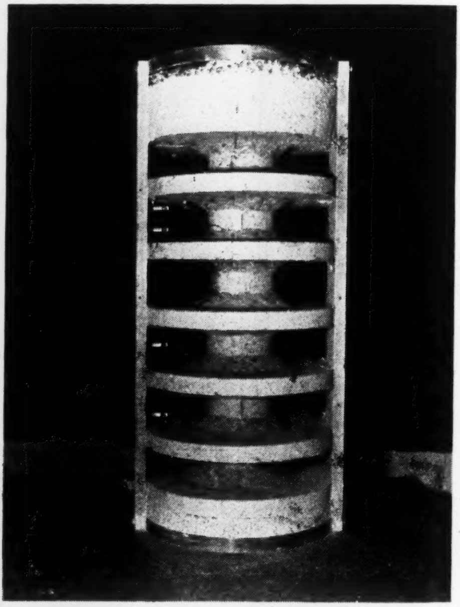

FIG. 4.—Creep Furnace, Shell and Loose Fill Insulation Removed.

spot welded to the specimen at top, bottom, and center. A period of 24 hr is normally allowed to get to temperature, during which time manual adjustments of the small autotransformers are made to obtain a temperature difference along the specimen gage length of less than $\frac{1}{2}$ F.

The construction of the furnace is shown in Fig. 4, with the shell and the loose fill insulation removed. It is 26 in. long and 11 in. in outside diameter. Direct exposure of the control thermopile and the specimen to radiation from the heater windings gives good response to changes in furnace voltage. The furnace winding is divided into 3 zones, and the distribution of power to the top and bottom zones is manually adjusted by small autotransformers. Figure 4 shows the method of installation of the convection stops in the insulated space around the heater elements of the furnace. They also serve to align the heater elements with the shell of the furnace.

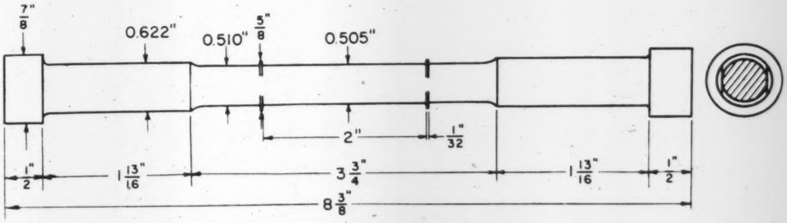

FIG. 5.—Creep Rupture Specimen.

Two types of extensometers have been used, both of which are actuated by rods extending below the furnace. Attachment of the rods to the specimen is made by means of two $\frac{1}{32}$-in. thick collars machined on the specimen at the 2-in. gage points and flattened on two sides as shown in Figs. 5 and 6. Tests of tension specimens, strained 20 per cent, have shown the reduction of area at points adjacent to the collar to be about 92 per cent of that in the middle of the gage length.

Compensation for thermal expansion is achieved by matching the expansion coefficient of the part of the extensometer rod which is alongside the specimen to that of the specimen. It was found that Nichrome V rods compensated for about 90 per cent of the thermal expansion of the carbon steel specimen when heated to 900 F; hence, with good temperature control in the room and in the furnace, the system has been quite satisfactory.

The extensometer for large elongations

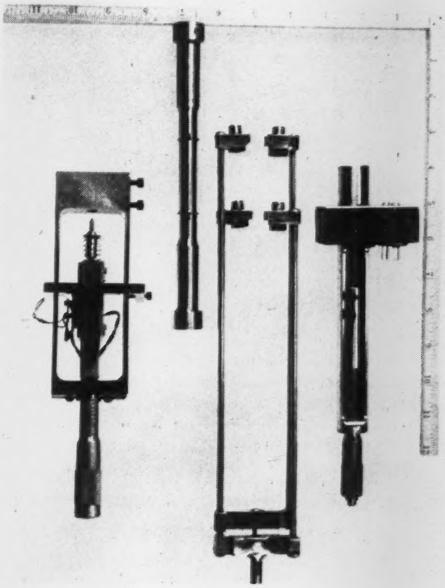

FIG. 6.—From Left to Right—Extensometer with Resistance Type Transducer, Specimen, Extensometer Rods, and Differential Transformer Extensometer.

uses a differential transformer pickup. Figure 6 illustrates the mounting provided for the coil and for the repositioning and calibrating micrometer. The differential transformer core is carried on the inner rod, connected to the upper gage point. When the core has traveled its full range, and the pen of the recorder as well, the micrometer is manually adjusted to reposition the core in the coil and by so doing to reposition the recorder pen. Full scale on the recorder is chosen to correspond to either 0.3 in. or 0.03 in. elongation. With the smaller range, it is sensitive to 0.000050 in. elongation.

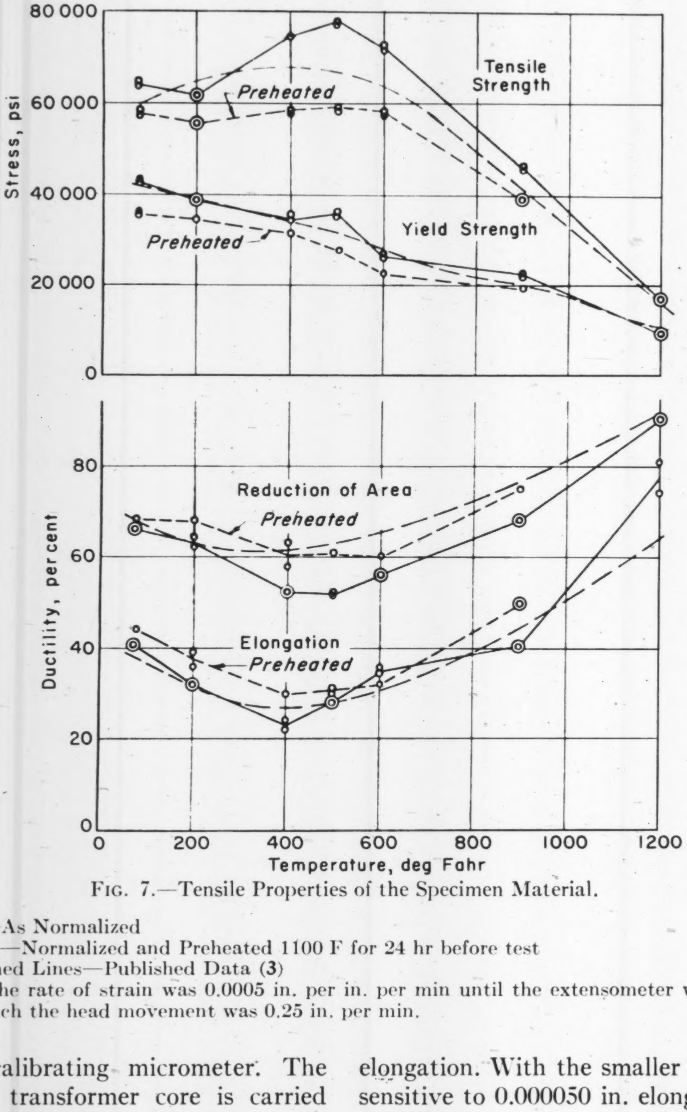

FIG. 7.—Tensile Properties of the Specimen Material.

Solid Lines—As Normalized
Dotted Lines—Normalized and Preheated 1100 F for 24 hr before test
Smooth Dashed Lines—Published Data (**3**)

NOTE.—The rate of strain was 0.0005 in. per in. per min until the extensometer was removed, following which the head movement was 0.25 in. per min.

The second type of extensometer pickup is an unbonded resistance wire device consisting of a complete four arm bridge of strain-sensitive wire. This pickup produces full scale on the recorder for 0.001 in. elongation. Sensitivity of this system is about 0.000005 in. elongation.

## DESCRIPTION OF TEST SPECIMENS

The specimen material conformed to the ASTM Specification for Carbon-Silicon Steel Plates (A201 Grade A, firebox quality). Chemical analysis of duplicate specimens from three locations in the plate gave the following average percentages: Carbon, 0.17; Manganese, 0.65; Phosphorus, 0.009; Sulfur, 0.022; and Silicon, 0.26. Further analyses of single specimens from three locations gave the additional results in per cent: nickel, 0.12; chromium, 0.08; molybdenum, 0.06; and copper, 0.22.

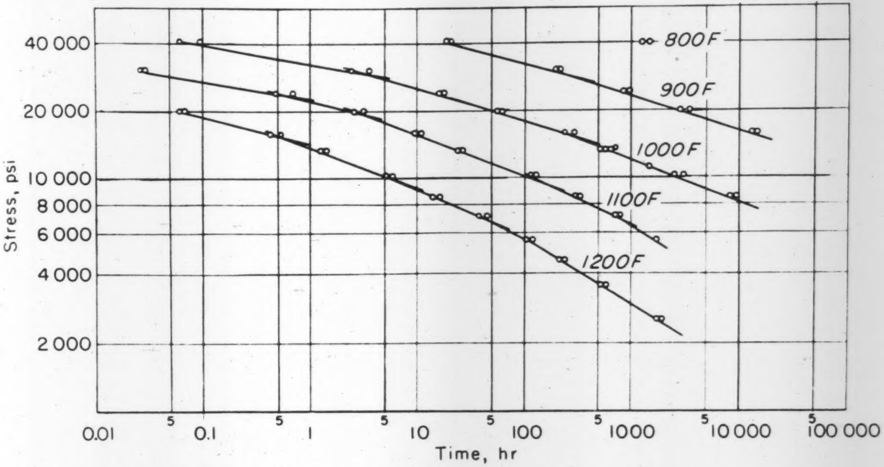

FIG. 8.—Stress-Time to Rupture at Five Temperatures.

The values reported for nitrogen and aluminum in the preprint of the paper were questioned by Mr. A. B. Wilder of the National Tube Division of the United States Steel Corp. He kindly offered to test a sample for these elements and reported the following percentages: Aluminum 0.006 and nitrogen 0.003. It is somewhat surprising that the residual alloy content totals almost 0.50 per cent, about twice that published in an industry survey (2) in 1944, which reported the following percentages: nickel, 0.05; chromium, 0.09; molybdenum, 0.02, and copper, 0.10. The supplier reported the deoxidation practice consists of the following additions to the ladle: 9.7 lb per ton of ferrosilicon, 2.0 lb per ton of silicon carbide, and 0.5 lb per ton of aluminum. No additions were made to the mold.

The specimen material was received in the form of a plate 1 in. thick by 6 ft wide by 14 ft long. The specimen blanks, 1 by 1 by 9 in., were sawed out with the 9-in. length in the direction of rolling. The blanks were normalized by holding 45 min at 1625 F, followed by air cooling. They were then machined to 0.505-in. diameter specimens as shown in Fig. 5.

Tensile properties of the specimen material after normalizing were determined at room temperature and at elevated temperatures up to 1200 F. These are plotted in Fig. 7 (solid lines), together with published values (3) for 0.12 to 0.17 per cent killed carbon steel, which are plotted as smooth dashed curves. The properties of this material conformed fairly well to the published data, although it exhibited a higher than normal tendency to strain aging as measured by the rise in ultimate strength and the decrease in ductility at about 500 F. For this material the ultimate strength at 500 F was 78,000 psi, 122 per cent of that at room temperature. The third set of curves in Fig. 7, showing the effect of an 1100 F, 24-hr heat treatment of the normalized material, will be discussed later.

*Rupture Life and Ductility:*

The conventional logarithmic stress-time to rupture plot is given in Fig. 8. Duplicate specimens were tested in all cases and were found to agree in rupture life within less than 15 per cent of the average of the pair of values. It was not possible to fit these data with one or two straight lines as is commonly done on plots of this type, except at 900 F. However, very short-time tests were not performed at this temperature. The tests at 1100 and 1200 F, particularly, show a stress rupture plot which has an almost continuous curvature, concave downward. The transition from transcrystalline to intercrystalline rupture is therefore not discernible on the stress rupture plot.

The proper way to explain the difference between our constant stress data and conventional constant load data, which is usually fitted with one or two straight lines, would be to run constant-load tests on our material. This has not yet been done, but the following hypothesis is offered as a possible explanation. The constant stress feature prolongs the rupture time for ductile specimens more than for brittle specimens, in comparison with constant-load tests. It will be shown that the specimens tested at 1100 and 1200 F and at intermediate test stresses had considerably greater elongation than the specimens tested at the lowest or the highest stresses. This hypothesis is consistent with the appearance of the data

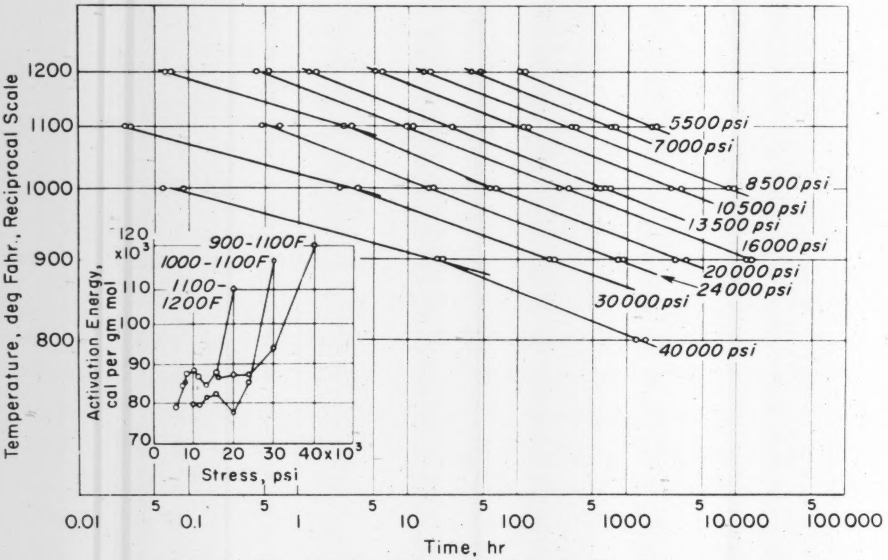

FIG. 9.—Temperature - Time to Rupture at Ten Stresses.

at 1000 F, which can be fitted by two or three straight lines fairly well and which did not exhibit a large variation in ductility with variation in rupture life.

The time to rupture data are replotted in Fig. 9 with temperature (reciprocal scale) as the ordinate rather than stress and with the logarithm of time as the abscissa as before. Conventional rate process theory (**4**) states that data of this type should plot as a family of parallel lines if the creep rupture process is governed by a single value of the activation energy for all of the data. The activation energy is inversely proportional to the slope of the family of lines. The data fulfill this criterion fairly well except for three very short-time tests. Computed values of the activation energy are shown in the inset. Most of the values given are in the range from 79,000 to 87,000 calories per gram mol, which is slightly higher than the value of 78,000 given by Dorn (**4**) for the self-diffusion of iron.

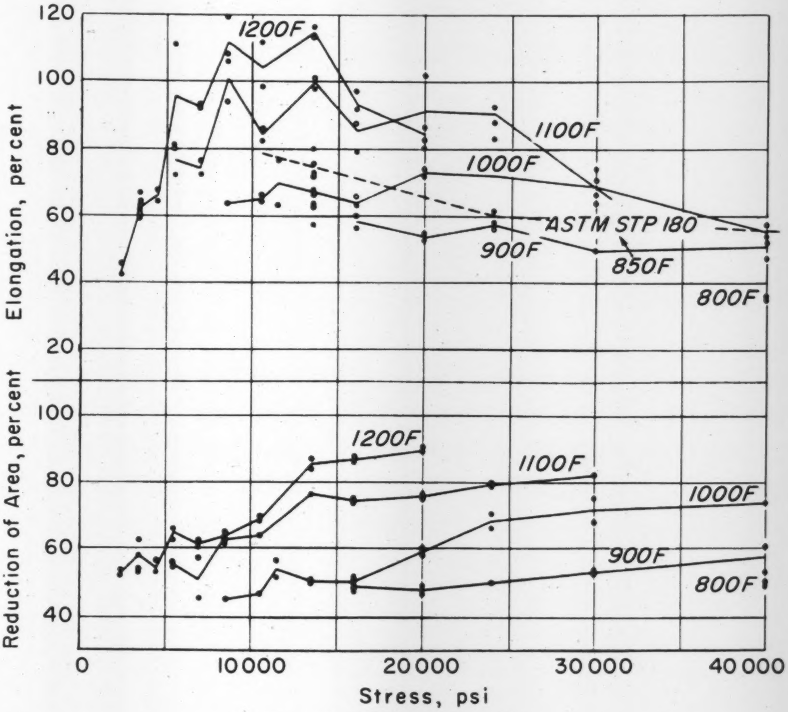

FIG. 10.—Rupture Ductility.

The elongation and reduction of area of the creep rupture specimens are plotted in Fig. 10. Published elongation data (**3**) for tests at 850 F seem to be roughly consistent with these data. The per cent elongation at 900 and 1000 F was nearly constant at all stresses, whereas at 1100 and 1200 F the elongation was greatest at the intermediate stress levels. No elongation values less than 37 per cent and no reduction of area values less than 45 per cent were observed. The rather low ductility which has been reported for killed carbon steel by the Timken Roller Bearing Co. (**5**) was not found in these tests even at times up to 10,000 hr. The drop-off in ductility of the long-time tests at 1100 and 1200 F is now believed to be due to oxidation. A few results of specimens tested in vacuum indicate a marked improvement in ductility and a slight improvement in rupture life.

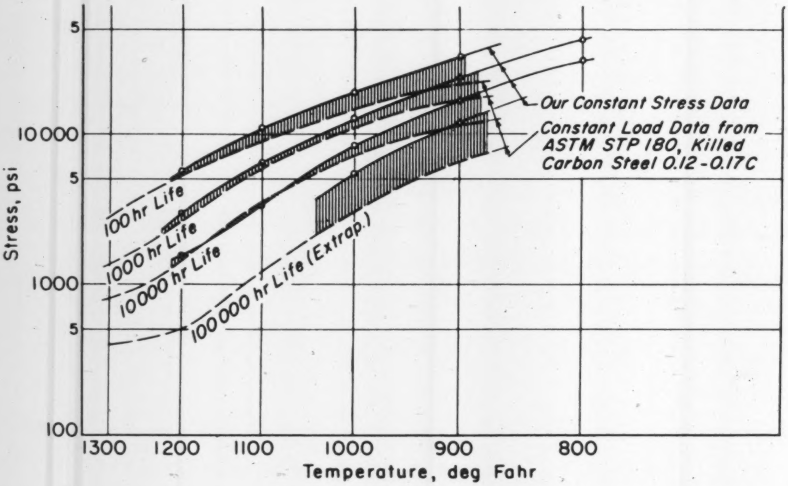

FIG. 11.—Comparison of Author's Rupture Data with Published Data.

*Comparison with Published Data:*

While a really good comparison of the constant-stress data with constant-load data is not possible without testing this material on constant-load equipment, it is interesting to compare it with published data for similar steels. This has been done in Fig. 11 which shows the stress for 100, 1000, 10,000, and 100,000 hr rupture life, plotted against the reciprocal test temperature. It can be seen that at 900 F the stress required for a given rupture life with this material and constant-stress equipment was roughly 50 per cent higher than that required for similar steels and constant-load equipment; at higher temperature, the difference was less. To see if this could be explained by the constant stress feature, the data shown in Fig. 12 were obtained on the variation of stress throughout the test for our equipment and for constant load equipment. Data from a 2000–3000-hr test at each of three temperatures are shown. The constant-load data are qualitative in that the "specimens" were assumed to have the same elongation and

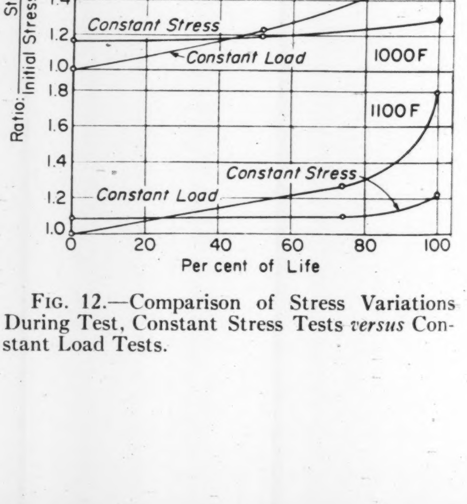

FIG. 12.—Comparison of Stress Variations During Test, Constant Stress Tests *versus* Constant Load Tests.

the same reduction of area as the constant-stress specimens.

The ordinate of the three graphs in Fig. 12 is the ratio of the stress at any instant (in either test) to the initial stress in the constant-load test. The constant-stress tests are shown starting the test at a stress ratio greater than 1.0, the value chosen being that required to produce the same rupture life as the constant-load test at that temperature (as given in Fig. 11). As the test progressed, the specimens under "constant" stress experienced a slight increase, less than 3 per cent midway in the test and less than 9 per cent at fracture. By comparison, the constant-load conditions would have caused an increase of stress of about 20 per cent midway in the test and 50 to 80 per cent at fracture.

TABLE I.—RATIO OF STRESS ON SPECIMEN AT RUPTURE TO INITIAL STRESS.
(Median values are given for the tests at each temperature, with the maximum and minimum values in parentheses.)

| Temperature, deg Fahr | Constant Stress Conditions | | Constant Load Conditions | |
|---|---|---|---|---|
| | Average Stress within the gage length | Maximum Stress at Fracture | Average Stress within the gage length | Maximum Stress at Fracture |
| | *1* | *2* | *3* | *4* |
| 900..... | 1.11 (1.08 to 1.14) | 1.44 (1.30 to 1.86) | 1.54 (1.47 to 1.60) | 2.00 (1.85 to 2.56) |
| 1000..... | 1.11 (1.07 to 1.15) | 1.46 (1.20 to 2.62) | 1.64 (1.53 to 1.84) | 2.13 (1.82 to 4.00) |
| 1100..... | 1.14 (1.07 to 1.18) | 2.30 (1.12 to 3.81) | 1.82 (1.66 to 2.09) | 3.70 (1.82 to 5.56) |
| 1200..... | 1.16 (1.06 to 1.20) | 1.69 (1.40 to 6.25) | 1.88 (1.41 to 2.20) | 2.70 (2.08 to 10.0) |

It appears that the constant-stress feature might explain the difference in stress for 1000 hr rupture life at 1000 F. The constant-stress specimen started life at a stress 17 per cent higher and ruptured at a stress 13 per cent lower than the equivalent constant load specimen. At 900 F, however, the stress in the constant-stress specimen was higher throughout the test, and at 1100 F it was lower for most of the test to give 1000 hr rupture life. It is concluded there must be an important difference in the behavior of this material and the 0.12 to 0.17 per cent killed carbon steel for which data have been published.

It should be explained that the stress referred to is the average within the gage length. The average cross-sectional area within the gage length was computed from the elongation of the specimen, assuming constant volume. The loads acting on the constant stress specimens midway in the test and at fracture were obtained from the over-all elongation between the button heads of the specimens, and the design equations of the equipment. The measurements of minimum cross-sectional area, required to compute the true stress at mid-life, were obtained from interrupted tests.

### *Performance of the Constant-Stress Feature:*

Further data on the constancy of stress for all test conditions are shown in Table I, which gives the ratio of the stress on the specimen at the end of the test to the initial stress on the specimen. The average stress within the gage length, computed as described above, is shown in column 1. The median values range from 1.11 for tests at 900 F to 1.16 for tests at 1200 F. This means that the average stress within the gage length was 11 to 16 per cent higher at the end of the test then it was at the beginning. But the area was not uniform along the gage length. Column 2 gives the maximum stress at fracture. The median values range from 1.44 at 900 F to 2.30 at 1100 F.

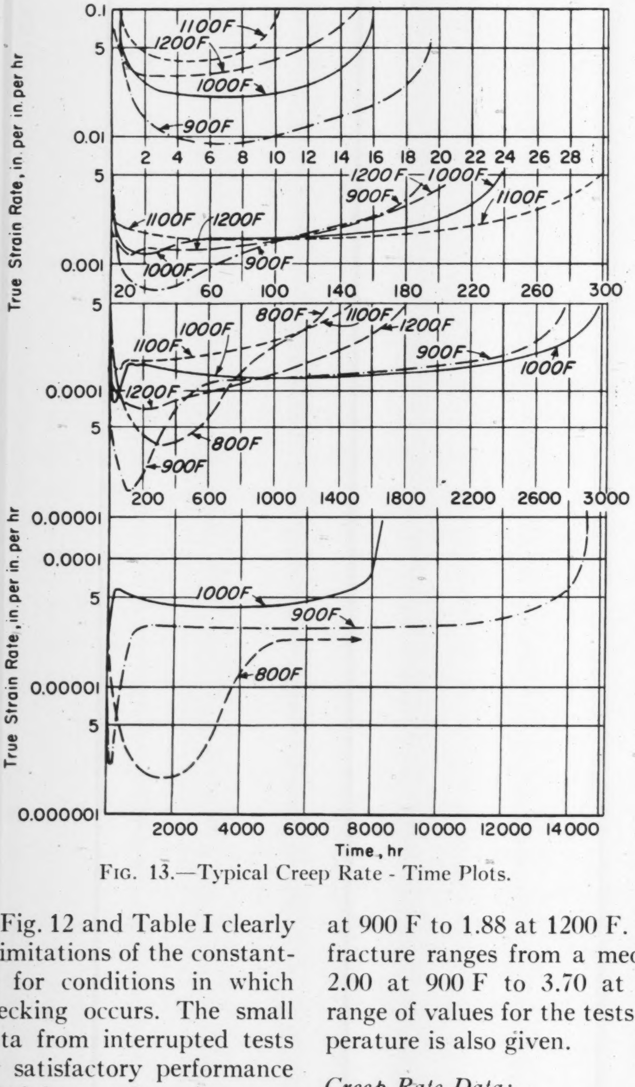

FIG. 13.—Typical Creep Rate - Time Plots.

The data in Fig. 12 and Table I clearly illustrate the limitations of the constant-stress feature for conditions in which appreciable necking occurs. The small amount of data from interrupted tests indicate fairly satisfactory performance in the first half of the test. Some improvement could be made by increasing the length of the reduced section of the specimen 2 to 3 per cent.

The constant-stress feature does produce a much better approach to the ideal test condition than does constant-load equipment, as can be seen from columns 3 and 4 of Table I. The range of median values of the average stress is from 1.54 at 900 F to 1.88 at 1200 F. The stress at fracture ranges from a median value of 2.00 at 900 F to 3.70 at 1200 F. The range of values for the tests at each temperature is also given.

*Creep Rate Data:*

The term "creep rate" is used to mean the true strain rate, computed by dividing the increment in elongation per unit of time by the specimen gage length at that instant. Typical creep rate-time plots are shown in Fig. 13, grouped according to rupture life. For example, the upper group shows a test of 10 to 20 hr duration at each temperature, but obviously not at the same stress.

The short-time tests exhibit the conventional shape, a rather flat minimum in the creep rate, occurring at about the midpoint of the test. By contrast, the long-time tests at 1100 and 1200 F show the minimum creep rates early in the test followed by a gradually increasing rate throughout most of the life of the specimen.

The long-time tests at 1000 F exhibit two minima in creep rate. The long tests at 900 and 800 F show a similar phenomenon, but the second minimum is only a leveling off in creep rate following recovery from the first minimum creep rate. The name given to the first minimum is "the dip." Experiments described below indicate it is caused by an unstable structural condition which gives good creep resistance for a limited time, following which the creep rate recovers from "the dip" and is subsequently governed by the characteristics of the "stabilized" material.

The variation of minimum creep rate with stress is shown in Fig. 14, both values plotted to logarithmic scales. The data at 900, 1000, and 1100 F were fitted fairly well with two straight lines; at 1200 F, three were required. For those tests in which there were two minima, the values for the first minimum fit the remainder of the data, the minimum creep rates at higher stresses, much better than do the values for the second minimum.

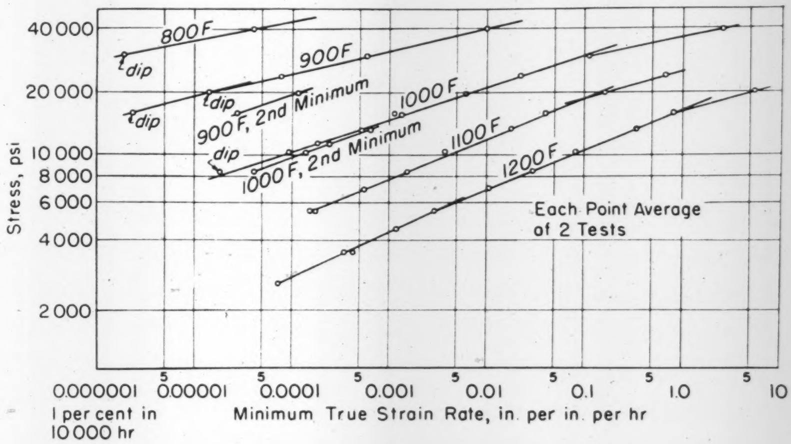

FIG. 14.—Stress *versus* Minimum Creep Rate at Five Temperatures.

The same creep rate data are replotted in Fig. 15, with temperature (reciprocal scale) as the ordinate rather than stress. Here, as in the plots of rupture life *versus* temperature, the slopes of the curves are the measure of activation energies for the creep processes which are going on. The bulk of the data shown in the inset are grouped around an average value of about 85,000 cal per gram mol, about the same as the values given in Fig. 9 for the temperature dependence of the rupture time. The values which are considerably higher than 85,000 are believed to result from a comparison of a test in which the structural condition causing the dip greatly affected the creep rate with a test in which it did not.

For those tests where there are two minima in the creep rate, both values are plotted. In this case, however, the rate which correlates with the remainder of the data is the second minimum rather than the first. In other words, it is the second minimum which correlates with high-temperature creep at that stress whereas it was the first minimum which correlated with high-stress creep at that temperature in the previous figure.

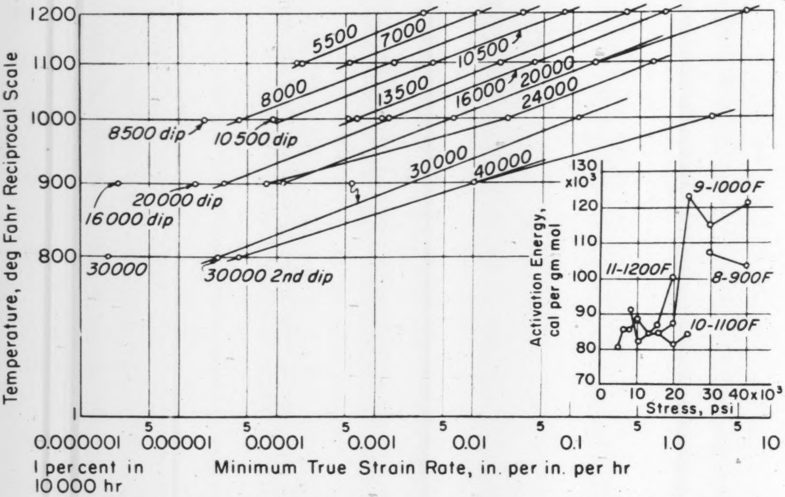

FIG. 15.—Temperature *versus* Minimum Creep Rate at Ten Stresses.

The form of the creep rate-time curves which exhibit two minima suggests that the results depend upon the net effect of two processes, one tending to increase the creep rate, the other tending to decrease it. Figure 16 is a schematic illustration of the argument for a case showing two minima of creep rate. The ordinate of Fig. 16 is the relative change in creep rate per unit of time. For example, if the creep rate is decreasing with time, strain hardening is taking place; the reverse is called strain softening. Curve *A* illustrates the form of the function for a creep rate-time relationship like those plotted in the upper part of Fig. 13. The strain hardening part of it can be explained in terms of dislocations, the motion of which becomes increasingly difficult as the test progresses. Curve *B* illustrates the form of a softening process suggested as the cause of the dip. Suppose for the purpose of this argument that the softening process goes to completion

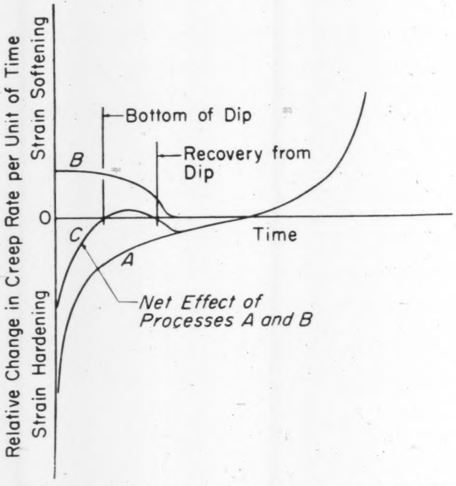
Relative Change in Creep Rate per Unit of Time
Strain Softening
Strain Hardening
Bottom of Dip
Recovery from Dip
B
0
C
A
Time
Net Effect of Processes A and B

FIG. 16.—Schematic Illustration of Strain Hardening Processes in Creep.

at a certain time, independent of the strain. Curve $C$ represents the net effect of processes $A$ and $B$. The time at which curve $C$ first crosses the time axis is the time for the creep rate-time curve to achieve the bottom of the dip. The second crossing of the time axis represents the time of intermediate maximum creep rate after the dip. Obviously, these times are affected by the form of curve $A$ as well as that of curve $B$, and the form of curve $A$ will vary almost directly with the time to rupture of the specimen. Hence, the shape of the dip shown in Fig. 13 varies greatly with stress as well as with temperature.

## EXPERIMENTS TO DEVELOP EXPLANATION FOR DIP

In one set of experiments, designed to explain the dip, the specimen was held at the test temperature or higher temperature for a period of time prior to the application of the load. Some results of tests designed to remove the dip in creep rate at 900 F and 20,000 psi (a 3000-hr test) are shown in Fig. 17. These show:

(*a*) 840 hr at 900 F barely removed the dip,

(*b*) 168 hr at 1000 F and 24 hr at 1100 F removed the dip to the same degree,

(*c*) 24 hr at 1200 F removed the dip,

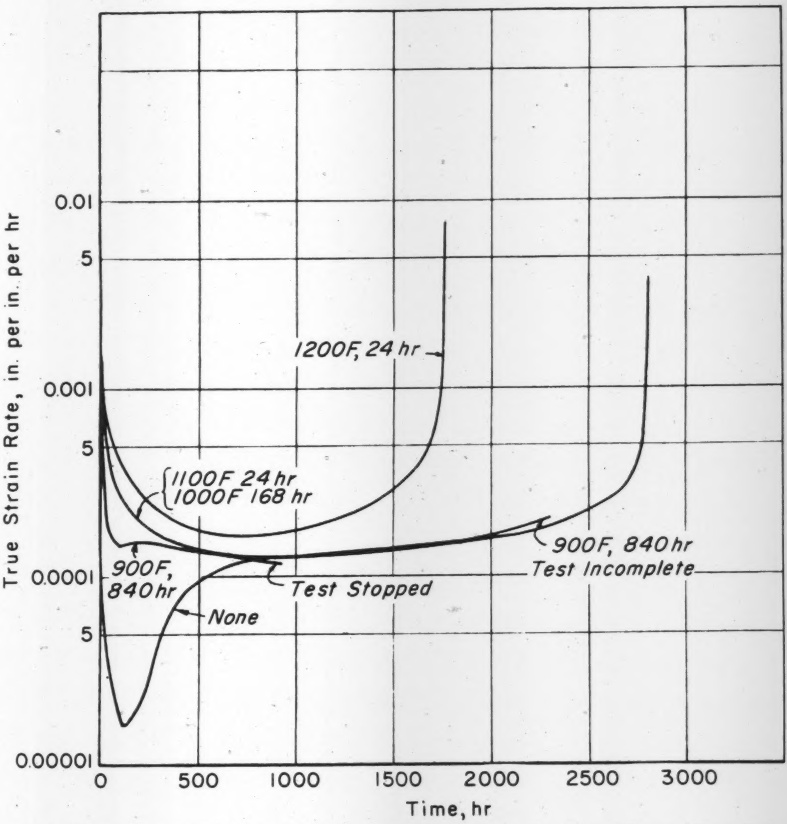

FIG. 17.—Effect of Certain No-Load Preheat Treatments on the Creep Rate - Time Relationship at 900 F and 20,000 psi.

increased the minimum creep rate 30 per cent above the rate established after the dip and shortened the life 50 per cent.

From the data obtained to date on the temperature-time preheating relationship for removal of the dip in tests at 900 F and 20,000 psi, it appears that the relationship is somewhere between that for diffusion of carbon in iron (36,600 cal per gram mol) and that for self diffusion of iron (78,000 cal per gram mol). Further study is required to interpret this finding.

In the first preheating experiments, the heat treatment was given in the furnace of the creep machine. This procedure oxidized the specimen surface appreciably in some cases and it also produced recovery of the surface layer of cold-worked metal left by the machining operations. Recovery was suspected as a possible cause of the dip and of its removal by the preheat treatment; and oxidation was a possible cause of the reduced life of the specimen preheated 24 hr at 1200 F. To check these possibilities, additional specimens were given the preheat treatment in bar stock form, following normalizing. The subsequent machining operations removed about $\frac{1}{4}$ in. of metal from around the reduced section of the specimens. The results were almost identical to those of the first preheating experiments. It was concluded that the recovery of cold-worked surface material is not a plausible explanation for the dip; nor is oxidation the cause for the reduced life of the specimen preheated 24 hr at 1200 F.

A possible mechanism for the transient softening process which causes the dip is the spheroidization of the pearlite lamellae as a result of time at temperature and of the ensuing strain. Studies of the microstructure of specimens tested at 900 F and 20,000 psi for one week, that is, to the bottom of the dip, showed no evidence of spheroidization. A similar study after 840 hr, that is, after recovery from the dip, also showed very little evidence of change in the form of the pearlite lamellae. Ruptured specimens under these same conditions did show considerable spheroidization of the carbides and tests at higher temperatures showed a very complete degree of spheroidization as well as coalescence of the carbide particles. Nevertheless, the lack of visible change in the microstructure after 840 hr makes this explanation of the dip unlikely. Also, as previously pointed out, the activation energy for the softening process induced by preheating is apparently higher than that for diffusion of carbon in iron, the governing process in spheroidization of a plain carbon steel.

Tension tests on specimens preheated at 1100 F for 24 hr after normalizing (a treatment which removed the dip) were made at several moderately elevated temperatures. The results are compared in Fig. 7 with similar data for specimen material which had received only the usual normalizing treatment. The preheating reduced the room temperature tensile strength 10 per cent and the yield strength 20 per cent. The preheating also greatly reduced the strain aging tendency; the ultimate strength at 500 F was only 2 per cent above that at room temperature, compared to a 22 per cent increase for the normalized material. The results of these tests indicate a possible correlation between the occurrence of the dip in creep rate shown by this material and the unusual degree of strain aging shown by the tensile data at 500 F. A possible explanation for the removal of the dip and also the removal of the peak in tensile strength at 500 F, produced by the 1100 F heat treatment, is that given by Miller (6) that nitrogen is removed from solution by chemical reaction with the aluminum present. Further work is planned.

## SUMMARY

Forty pairs of specimens of ASTM Specification A 201[3] killed carbon steel have been tested in constant-stress creep rupture equipment at five temperatures ranging from 800 to 1200 F and stresses chosen to give rupture lives ranging from 0.1 to 10,000 hr. The relationship between the logarithms of stress and time to rupture was not linear, particularly at 1100 and 1200 F, as it commonly is with constant-load equipment. This is possibly because the elongation varied with stress, and the constant stress feature tends to prolong the life of ductile specimens more than brittle ones, compared to constant load equipment. Even the long-time specimens showed good ductility, the lowest elongation observed being 37 per cent. The stress for a given rupture life was found to be considerably higher than published data for killed carbon steel, particularly at 900 and 1000 F. This could not be explained by the constant-stress feature of our equipment.

A study of the constancy of stress revealed that the average stress along the gage length rose 2 or 3 per cent during the first half of the test and from 11 to 16 per cent at rupture. The latter are median values for all specimens. Median values for the maximum stress at the time of rupture (at the fracture) ranged from 1.44 to 2.30 times the initial stress.

Studies of the variation in creep rate throughout the tests revealed the existence of two minima in long-time tests at 1000 F and below. It appears that the material was in an unstable structural condition which imparted unusually good resistance to creep at the beginning of these tests but which was eliminated by the lapse of time (or perhaps due to strain) during the test, allowing recovery from the first minimum, "the dip". It is presently believed to be a strain aging phenomenon.

A variety of heat treatments, following normalizing, were found to remove the dip. One of them, 24 hr at 1100 F, also reduced the room temperature tensile strength 10 per cent and practically eliminated the peak in tensile strength at 500 F, exhibited to an unusual degree by the normalized material. The temperature *versus* time-of-preheating relationship for removal of the dip indicates the process is not governed solely by the diffusion of carbon in iron, nor by the self diffusion of iron. Preheating the specimen in bar stock form produced the same effect as when done after assembly in the creep machine, indicating recovery of cold-worked metal on the surface of the specimen was not the cause of recovery from the dip. Microstructural evidence did not support the theory that spheroidization was the cause of the dip. The specific nature of the unstable structural condition causing the dip is still being studied.

*Acknowledgment:*

The author gratefully acknowledges the leadership of Mr. G. W. Watts, Director of Engineering and Engineering Research, and of W. M. Dudley, Associate Director of Engineering Research, Standard Oil Co. (Indiana) whose interest in creep rupture led to the establishment of the research facility described. He also wishes to acknowledge the craftsmanship of Mr. F. V. Fary, Research Assistant, who conducted the tests.

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[3] Tentative Specification for Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 54 T), 1955 Book of ASTM Standards, Part 1, p. 487.

## REFERENCES

(1) E. N. Andrade and B. Chalmers, "The Resistivity of Polycrystalline Wires in Relation to Plastic Deformation, and the Mechanism of Plastic Flow," *Proceedings*, Royal Society," Vol. 138, p. 348 (1932 (A)).

(2) J. D. Sullivan and A. E. Pavlish, "Residual Metals in Open Hearth Steel,"*Open Hearth Proceedings*, Am. Inst. Mechanical Engrs., pp. 210–219 (1945).

(3) "The Elevated Temperature Properties of Çarbon Steels," Am. Soc. Testing Mats. (1955). (Issued as separate publication, *ASTM STP No. 180.*)

(4) John E. Dorn and Lawrence A. Shephard, "What We Know About Creep," Symposium on Effect of Cyclic Heating and Stressing on Metals at Elevated Temperatures, Am. Soc. Testing Mats. (1954). (Issued as separate publication, *ASTM STP No. 165.*)

(5) "Digest of Steels for High Temperature Service," Timken Roller Bearing Co., Fifth Edition (1946).

(6) R. F. Miller, "The Strength of Carbon Steels for Elevated Temperature Applications," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 964 (1954).

# DISCUSSION

MR. JOSEPH MARIN.[1]—My question relates to the use of the creep data in design.

I can see the fundamental value of keeping the stress constant. But is it not a fact that the main application of such data would be more concerned with defining failure based on the beginning of instability or necking?

Has the predicted strain at the beginning of instability or necking been compared with the actual value?

MR. PRYOR N. RANDALL (*author*).—To answer the first question, we were motivated in the beginning simply by a feeling that creep rate data were going to waste in a rupture test; hence, we chose to record creep rates and to add the constant stress feature to facilitate their interpretation. As you suggest, this should permit us to study the onset of third stage creep as evidence of weakening of the material brought on by the preceding strain and time at temperature. The equipment is not perfect for this study because the weakening of the material causes instability and local necking, which the constant stress feature does not fully correct for. However, the study suggested should be worthwhile.

Another reason for building constant stress equipment is that we plan to run tests in which the load and temperature are varied during the test. By eliminating the stress variation due to changes in specimen cross-section we hope to simplify the interpretation of the results.

In answer to the second question, we have not made any theoretical predictions of the strain at the beginning of instability or necking.

MR. HOWARD CROSS.[2]—I am a little confused. I have been trying to figure out why the log-log plot should fall away from a straight-line relationship. In a constant-stress test, the average stress is lower during the life of a ductile material than when using constant load. I think the constant-stress condition would tend more toward the straight-line relationship than away from it.

MR. RANDALL.—As mentioned, the average stress imposed on a specimen during its life was lower in the constant stress equipment than it would have been in a constant load machine. Therefore, the comparable stress-rupture plot for constant load data should lie to the left of the constant stress plot, that is, a specimen tested at the same nominal stress would rupture in a shorter time in constant load equipment. The difference in rupture time would be greatest for the most ductile specimens, which were those tested at the intermediate stress level. Therefore, as I picture it, the constant load data should appear as the chord of the arc formed by the constant stress plot.

MR. A. B. WILDER.[3]—I notice that the

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[1] Professor and Head, Department of Engineering Mechanics, College of Engineering and Architecture, Pennsylvania State University, University Park, Pa.

[2] Assistant Technical Coordination Director, Battelle Memorial Institute, Columbus, Ohio.

[3] Chief Metallurgist, National Tube Division, U. S. Steel Corp., Pittsburgh, Pa.

aluminum content is 0.03 per cent which to me would indicate a fine grain steel, whereas the amount of aluminum added was 0.5 lb per ton, which would indicate coarse grain steel. There seems to be a little inconsistency there.

I should like to ask a question: How do you apply this constant stress data to the performance, for example, of a carbon steel tube in service where there would be swelling. What is the application of this type of data to the performance, say, of a tube in service particularly where it results in failure due to swelling?

MR. RANDALL.—To answer the first question, the grain size was fairly fine—between 7 and 8 ASTM number.[4] (In a later conversation, Mr. Wilder kindly offered to check the aluminum content for me. The result, 0.006 per cent is the value now given in the paper.)

In answer to the question concerning the application of constant stress data to a tube; as has been pointed out, the diameter of the tube increases and its wall thickness decreases, so the stress goes up faster than it does in a constant load creep test because of the two effects. We would have to do a step-by-step calculation of stress *versus* creep rate on the tube, assuming a number of increments of strain and a value for the total ductility which the tube was going to exhibit and compute the time required to use up that ductility.

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[4] Classification of Austenite Grain Size in Steels (E 19 – 46), 1955 Book of ASTM Standards, Part 1, p. 1602.

# MICROHARDNESS TESTING AT HIGH TEMPERATURES*

By J. H. Westbrook[1]

## Synopsis

This paper describes the construction, operation, and characteristics of a hot-hardness tester of modified Bergh design with a temperature range of 20 to 1500 C and a load range of 20 to 900 g. The problems encountered in this type of test, particularly with respect to hard, brittle materials, are discussed in some detail. Typical results are shown, and examples of the application of this test described.

The indispensability of tests at or above projected service temperatures has been well established in numerous investigations. However, the experimental difficulties in running the conventional type of mechanical-property tests at high temperatures are considerable, and the time required per specimen may be unreasonably long. Attention has therefore often been given to hot-hardness testing as a simplifying procedure. Its principal advantages are the simple shape of the specimen required and the large number of specimens that can be measured in a given time. The correlation of hot-hardness data with those of other conventional high-temperature tests has been adequately demonstrated (**1,2,3**),[2] although the detailed interpretation of these data may not be clear. The hot-hardness test is particularly suited to studies of the behavior of hard, brittle materials such as ceramics and intermetallic compounds. With such materials, it is frequently very difficult if not impossible to machine a shape of complex geometry as for a tension specimen. However, a suitable surface for hardness testing can usually be obtained with ease by grinding a flat face, followed by diamond polishing. Further, the problem of obtaining perfect uniaxial loading, preeminent in the tension testing of brittle materials, is largely eliminated in the hot-hardness test. The possibility of obtaining data from relatively small specimens afforded by the hardness test may also be significant in cases in which it is not convenient to prepare large pieces of the test material. Notwithstanding these advantages, hot-hardness testing is beset with several problems of its own, which are discussed below. In general, these problems become prominent with high temperatures, low loads, and hard, brittle materials.

If discussion is restricted to the indentation type of hardness measurement, we may still consider two broad classes of tests—the static tests and the dynamic. These are chiefly differentiated by the difference in rate of loading and in duration of load. In the dynamic type, one seeks to minimize the effects of time through an impact type of test but with the possible disadvantage of a high loading rate. In the static type, the rate of loading is quite moderate and the duration of load may be from a few seconds to an hour or more. As has been pointed out (2), a higher degree of standardization has been achieved with the static method than with the dynamic method. In addition, for brittle materials, a slow loading rate is virtually prerequisite to obtaining useful data. Hence, the static type of test is to be preferred.

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* Presented at the Sixtieth Annual Meeting of the Society June 16–21, 1957.

[1] General Electric Research Laboratory, Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 894.

Similar considerations led to the choice of a micro- over a macro-hardness tester. With very tiny impressions, a large amount of data can be obtained from a small specimen. Further, in hard, brittle materials, satisfactory impressions, free of shattering, can be obtained only with very light loads (100 g or less). To summarize, then, our object was to construct a micro hot-hardness tester particularly adapted to hard, brittle materials and having a high operating-temperature range (1500 C was assumed to be an attainable maximum).

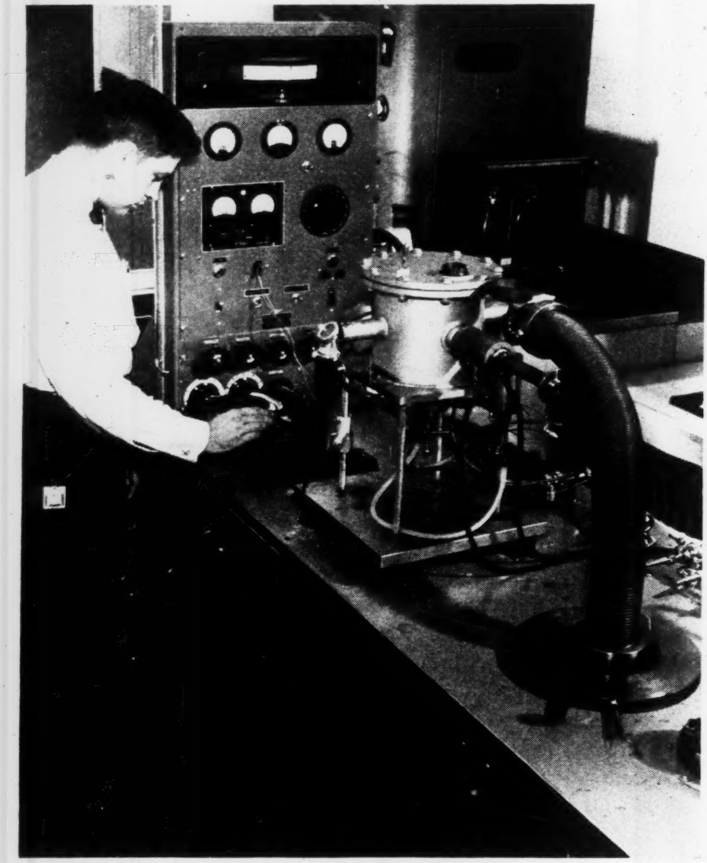

FIG. 1.—Photograph of Test Equipment.

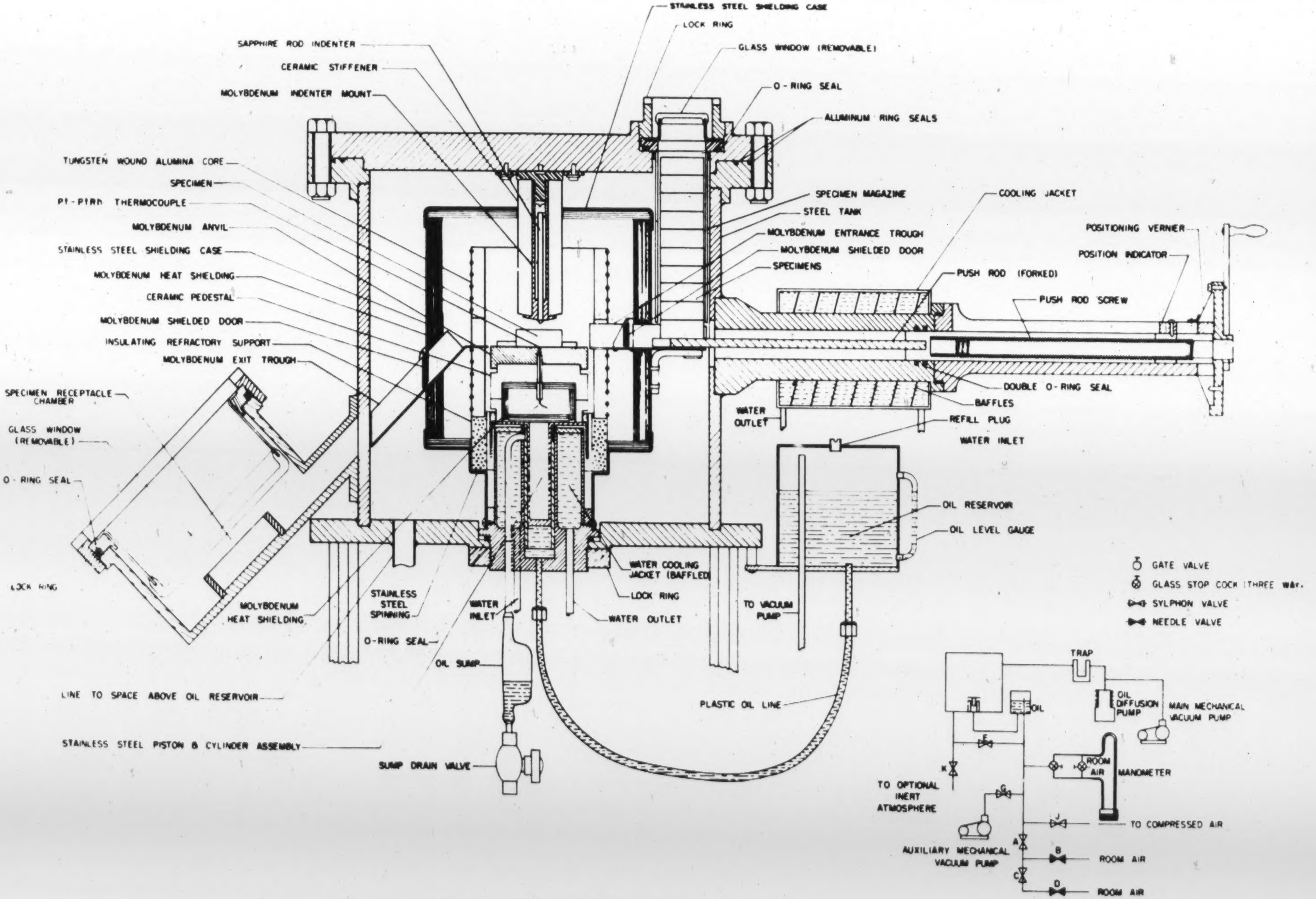
SAPPHIRE ROD INDENTER
CERAMIC STIFFENER
MOLYBDENUM INDENTER MOUNT
TUNGSTEN WOUND ALUMINA CORE
SPECIMEN
Pt-PtRh THERMOCOUPLE
MOLYBDENUM ANVIL
STAINLESS STEEL SHIELDING CASE
MOLYBDENUM HEAT SHIELDING
CERAMIC PEDESTAL
MOLYBDENUM SHIELDED DOOR
INSULATING REFRACTORY SUPPORT
MOLYBDENUM EXIT TROUGH
SPECIMEN RECEPTACLE CHAMBER
GLASS WINDOW (REMOVABLE)
O-RING SEAL
LOCK RING
MOLYBDENUM HEAT SHIELDING
STAINLESS STEEL SPINNING
O-RING SEAL
LINE TO SPACE ABOVE OIL RESERVOIR
STAINLESS STEEL PISTON & CYLINDER ASSEMBLY
OIL SUMP
SUMP DRAIN VALVE
STAINLESS STEEL SHIELDING CASE
LOCK RING
GLASS WINDOW (REMOVABLE)
O-RING SEAL
ALUMINUM RING SEALS
SPECIMEN MAGAZINE
STEEL TANK
MOLYBDENUM ENTRANCE TROUGH
MOLYBDENUM SHIELDED DOOR
SPECIMENS
COOLING JACKET
POSITIONING VERNIER
POSITION INDICATOR
PUSH ROD (FORKED)
PUSH ROD SCREW
DOUBLE O-RING SEAL
BAFFLES
WATER OUTLET
REFILL PLUG
WATER INLET
OIL RESERVOIR
OIL LEVEL GAUGE
WATER COOLING JACKET (BAFFLED)
LOCK RING
WATER INLET
WATER OUTLET
TO VACUUM PUMP
PLASTIC OIL LINE
GATE VALVE
GLASS STOP COCK (THREE WAY)
SYLPHON VALVE
NEEDLE VALVE
TRAP
OIL
OIL DIFFUSION PUMP
MAIN MECHANICAL VACUUM PUMP
ROOM AIR
MANOMETER
TO OPTIONAL INERT ATMOSPHERE
TO COMPRESSED AIR
AUXILIARY MECHANICAL VACUUM PUMP
ROOM AIR
ROOM AIR
A
B
C
D
E
F
G
H
J
K

FIG. 2.—Schematic Drawing of Test Equipment.

## PREVIOUS HOT-HARDNESS TEST EQUIPMENT

A survey of the literature revealed no equipment that met our specifications. The conventional hot-hardness apparatus (see, for example, References 1–6) had several defects: no control of loading rate, high loads, low temperature range. However, one design described by Bergh (7) overcame most of these disadvantages and appeared to be adaptable for operation in the 1500 C range. The design ultimately adopted was arrived at following a series of modifications of the Bergh design. The following description covers the main features of the instrument. Further details of design and construction may be found in a paper by Bergh (7).

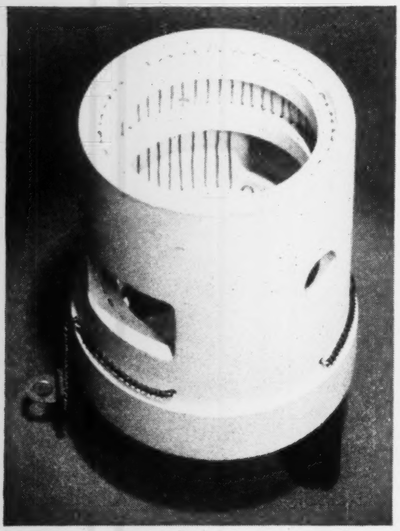

FIG. 3.—Furnace Core, Showing Vertical Internal Winding (Three Parallel Windings of 0.015-in. Tantalum).

## DESCRIPTION OF APPARATUS

*Principal Features:*

A photograph and schematic drawing of the equipment are shown in Figs. 1 and 2. In common with most indentation-type testers, this instrument consists essentially of an anvil that supports the specimen, a fixed indenter, a means for positioning the specimen beneath the indenter, and a loading system. Indentations are made with a known load, and their dimensions measured subsequently by microscopic methods. Hardness numbers are then calculated from these data by employing a formula appropriate to the geometry of the indenter used. The high-temperature range desired dictates the use of vacuum or inert atmosphere for protection of sample, indenter, and furnace winding. Bergh, therefore, found it convenient to use a hydraulic loading system with air as the primary loading medium acting on a column of oil (see further below).

Specimen feed was accomplished by means of a screw-driven molybdenum push-rod acting through an appropriate O-ring vacuum seal. This arrangement gave an accuracy of position well within ±0.001 in. Specimens are introduced into a removable magazine suspended from the top cover of the tester. As needed, they can then be "dealt from the bottom of the deck" onto a trough and thence to the anvil by means of the push-rod. After all the desired impressions have been made in a given sample, it can be pushed off the anvil and caused to slide down the exit trough and into a receptacle chamber, external to the main test chamber. Both the specimen magazine and receptacle chamber are provided with viewing windows and O-ring seals. Specimens are in the form of small disks, which may vary in dimension from about $\frac{1}{2}$ to 1 in. in diameter and from $\frac{1}{4}$ to $\frac{1}{2}$ in. in thickness. Specimens smaller than these dimensions and of other shapes can be handled through the use of appropriate mounting jigs. The number of specimens that can be accommodated in the magazine varies from about 8 to 16, depending on their thickness.

The testing chamber is evacuated through a large-diameter flexible metal hose connected to the working plate of a general-purpose vacuum system. The vacuum system itself consists of a Distillation Products Inc. oil diffusion pump backed up with a Welch duo-seal mechanical pump. A liquid-nitrogen trap and Pirani and McLeod gages complete the system. With this set-up, a vacuum of better than 0.2$\mu$ was maintained at all temperatures.

*Furnace and Associated Components:*

A V-10 General Radio variac driving a 2:1 current transformer provided a maximum input power to a resistance heater of 1 kw at 20 amp and 50 v, with stepless control over the full range. In our first furnace design, 0.030-in. diameter tungsten wire was helically wound on an alumina core. Thermal insulation was furnished by radiation heat-shielding of concentric layers of prick-punched sheet molybdenum. The necessity of providing holes in both the core and shielding for the introduction and extraction of the specimen as well as for a sight port considerably lowered the thermal efficiency of the design, particularly at high temperatures. The addition of shielded trap doors in both the exit and entrance troughs helped only slightly. A later core design therefore used an internal vertical winding of 0.015-in. diameter tantalum wire. This design provided much better wire coverage in the vicinity of the specimen, as well as the elimination of the insulation between wire and specimen, and therefore resulted in significantly better efficiency at high temperatures. A temperature of 1500 C could be maintained with 900 w. A wound core of the improved design is illustrated in Fig. 3. Temperatures were measured by means of a platinum-platinum rhodium thermocouple peened into the surface of the molybdenum anvil on which the specimen rests. This couple was connected to insulated binding posts mounted on the base plate of the heater and thence, through this base, to the control panel with compensating leads, sealed to the vacuum system with wax. At the control panel, a double pole-double throw switch permitted connection of the thermocouple to either an on-off controller or a hand-operated potentiometer.

*Loading System:*

As shown in Fig. 2, the anvil rests upon a hollow ceramic cylinder which in turn is carried on the piston of the water-cooled hydraulic system. Diffusion-pump oil (Octoil 8) is used as the hydraulic medium. The loading operation is as follows. The space above the oil in the oil reservoir is separately evacuated by an auxiliary mechanical pump. Adjustment of a controlled air leak in this line varies the fraction of an atmosphere applied above the oil in the reservoir and thus transmitted to the piston. A small fraction of the available pressure range is consumed by the force required for raising the piston, ceramic support, anvil, and specimen. The balance gives a working range of about 20 to 900 g. Loads are measured with a mercury manometer previously calibrated with balance weights on the piston. A plot of load *versus* manometer reading gave a straight line of slope 0.0560 cm per g. Since a mercury manometer can be read without great difficulty to about ±0.1 mm, the accuracy of load measurement is about ±1 per cent at the low end of the range and ±0.02 per cent at the high end of the range. The effect of piston friction on accuracy of loading was determined at both room and elevated temperatures. A condition was ultimately achieved in which, with rare exception,

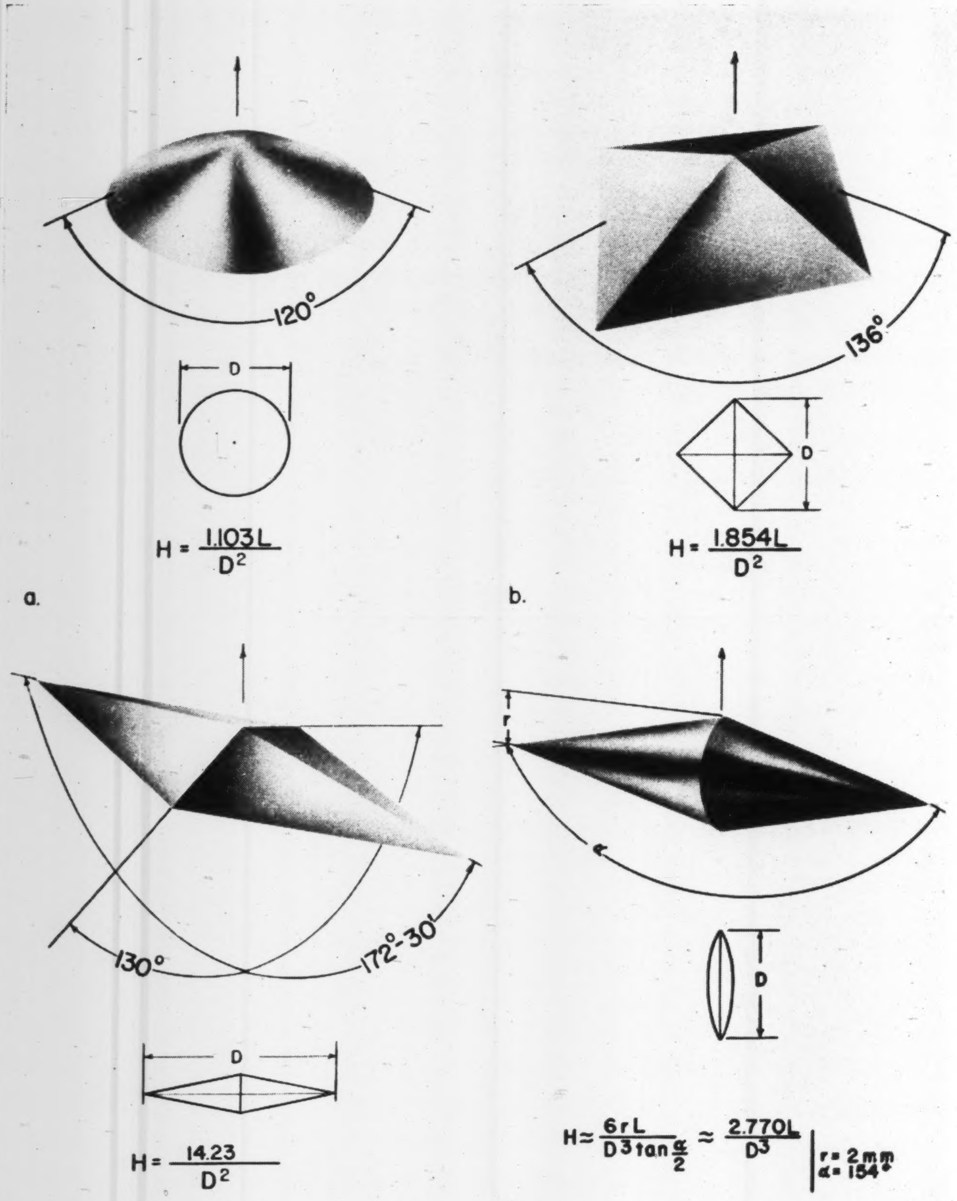
120°
D
$H = \frac{1.103L}{D^2}$
a.
136°
D
$H = \frac{1.854L}{D^2}$
b.
130°
172°-30'
D
$H = \frac{14.23}{D^2}$
c.
r
α
D
$H \approx \frac{6rL}{D^3 \tan\frac{\alpha}{2}} \approx \frac{2.770L}{D^3}$
r = 2 mm
α = 154°
d.

FIG. 4.—Indenter Geometry: (*a*) Cone, (*b*) Square-Based Pyramid, (*c*) Knoop, (*d*) Double Cone.

the friction effect was within the accuracy of manometer reading. Valving was provided for both the manometer and loading system, so that, when desired, an inert atmosphere could be substituted for the vacuum in the testing chamber with auxiliary pressure supplied by compressed air rather than by atmospheric pressure. The system was designed and built with two sets of two valves each for control of loading. Each set consisted of a quick-opening vacuum valve and a geared fine needle valve; one set was for the preload to raise the specimen against the indenter, the other for applying the load to the specimen. It was originally thought that preload and load valves could be set initially at the desired values for a given run, and, following this adjustment, a large number of impressions might be made expeditiously by manipulation of only the quick-open valves. This idea did not work out in practice; the small but unavoidable change in valve characteristics with time gave a variation in load relative to the total load which could not be disregarded except at high loads. Consequently, preload and load values are set for each indentation by manual adjustment of the needle valves.

### *The Indenter:*

The design of the indenter and its mount and the selection of the indenter material proved to be one of the most difficult problems encountered. It was felt desirable to maintain the temperature of the tip of the indenter close to the temperature of the specimen. Attempt was made to achieve this control by minimizing the cross-section of metal in the indenter mount and by using materials of low thermal conductivity to insulate the tip. At the same time, the mount was required to be fairly rigid at all temperatures in order to maintain axiality with respect to the specimen. It is believed that these goals were realized to a degree sufficient for practical operation.

Choice of indenter geometry is a particularly important problem. There are three forms that have found considerable use in microhardness testing—the square-based pyramid, the cone, and the rhombohedral-based pyramid or Knoop shape.[3] The ball is not favored because of the change in geometry with increasing depth. Recently, a fourth form has been introduced by Grodzinski—the so-called double cone (**8,9**). These various shapes are illustrated in Fig. 4, which also shows the specifications and pertinent formulae for computing the hardness number.[4]

The square-based pyramid has been widely used because of the difficulty in cutting diamonds to spherical or conical shapes as a result of their rather pronounced anisotropy. On the other hand, as compared with the cone, the pyramid introduces undesirable stress concentrations at the corners of the impression, which lead to cracking in brittle materials. In addition, it is extremely difficult to cut a pyramidal indenter to a truly sharp point, since each facet must be cut and polished separately. An error in the amount of material removed on any facet will give a ridge rather than a point. The sharpest point commercially possible to date is about $0.5\mu$.[5]

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[3] The triangular base pyramid proposed by the Russians (**7a**) has been little used elsewhere and appears to offer no significant advantage over the other types except ease of manufacture.

[4] The pyramid and cone numbers are calculated on the basis of contact surface area and are usually expressed in terms of kg per sq mm. The Knoop and double-cone hardness numbers are based on the unrecovered projected area, but are expressed in the same terms. An approximation is involved in the case of the double cone in that the bounds of the impression are taken to be parabolae rather than hyperbolae, as is actually the case.

[5] A paper by Campbell *et al* (**10**) contains a valuable discussion of accidental variations in tip geometry.

The Knoop indenter shown in Fig. 4(*c*) was the result of an effort by a group at the National Bureau of Standards to develop an indenter suitable for very small loads and brittle materials (**11**). This objective was accomplished by modifying the pyramid to more of a wedge shape through the use of a rhombohedral base. With the angles specified, the length-width-depth ratios for the impression are about 30:4:1. Elastic recovery consequently takes place principally in the direction of the shorter diagonal and scarcely at all in the direction of the long diagonal. Grodzinski's double cone bears the same relation to the Knoop that the cone bears to the square-base pyramid. The length-width-depth ratios for the standard form of his design are about 150:9:1. As a consequence of the narrower and shallower indentation and the elimination of two points of stress concentration, cracking in brittle materials is substantially reduced. It is also claimed that this indenter can be used with higher loads without cracking, so that the effect of load on hardness, normally encountered, is eliminated. However, unlike the other shapes shown in Fig. 4, the double-cone indenter does not maintain similarity of indentations with increasing load. The width-to-depth ratio remains constant, but the length-to-depth ratio varies widely with the amount of penetration from about 25:1 to 800:1 for the normal range of hardness values and loads.

TABLE I.—ROOM-TEMPERATURE MICRO-HARDNESS OF SOME POTENTIAL INDENTER MATERIALS.

| Material | Melting Point, deg Cent | Room-Temperature Micro-hardness $K_{100}$, kg per sq mm |
|---|---|---|
| Diamond | 3700 | 6000 to 8000 |
| $B_4C$ | 2350 | 2200 to 5000 |
| SiC | 2700 | 2000 to 4200 |
| $TiB_2$ | 2980 | 3400 |
| TiC | 3250 | 2350 to 3200 |
| Sapphire ($Al_2O_3$) | 2050 | 950 to 2250 |

Choice of indenter material involved several factors: high hardness (especially at elevated temperatures), stability, chemical inertness, and availability. Unfortunately, data on the high-temperature hardness of the very hard materials can only be produced by just such a machine as is now being described. Therefore, the only available guide is the room-temperature hardness. Such data, taken from the literature, for a few of the potential indenter materials are given in Table I.

Diamond, the first choice on the basis of hardness, fails to meet several of the other requirements. The diamond form of carbon is thermodynamically unstable, even under ordinary atmospheric conditions, and it is only because of the extremely slow kinetics of the reaction to graphite, the stable form, that we enjoy the beauty and utility of the diamond. At temperatures in the 900 to 1000 C range, in most instances the graphitization reaction proceeds at an appreciable rate, thus putting an upper limit on the operating range. Coincidentally, this range is about the same maximum temperature imposed by the conventional diamond-mounting alloys (**1, 2**), owingto softening and volatilization *in vacuo.* A further difficulty arises from the high chemical reactivity between the diamond and certain of the materials to be tested. For example, it has been found (**12,13**) that repeated indentations with diamond in titanium-rich samples even at room temperature results in a built-up layer of titanium metal on the indenter tip. While such films reportedly can be scraped off without apparent damage to the surface of the diamond, similar films built up during high-temperature operation are apparently much more adherent. Tests in this laboratory at temperatures up to 850 C on high-purity chromium, another strong carbide former, resulted in considerable deterioration of the diamond surface. Garafalo *et al* (**2**) had similar experience with stainless steels at 815 C.

Only two other materials are known to have been used successfully as high-temperature indenters: cemented carbides (**3**) and sapphire (**2,5**). Cemented materials are obviously unusable at temperatures above the softening point of the binder, nor are they well suited to micro-work. Sapphire, on the other hand, is quite attractive as an indenter material. Rods and various other shapes of the synthetic material are available at moderate cost. The melting point is reasonably high, and the compound is stable and nonreactive. It has been used successfully on chromium-bearing alloys where diamond had proved too reactive (**2**). One paper (**5**) reports the use of sapphire indenters at temperatures as high as 1900 C. Unfortunately, these indenter points are rather fragile as compared with diamond, and further, as can be seen from Table I, their hardness is too low for them to be of use in testing many of the hard compounds such as the carbides, borides, and nitrides. Some sapphires were ground, polished, and mounted for use with this equipment, but it was realized that further search would have to be made for an accurate high-temperature indenter for harder materials.

Attempts were therefore made to fashion indenters from other hard refractory materials. Samples were obtained[6] of small single crystals of silicon carbide, boron carbide, and titanium carbide. The titanium carbide crystals proved to be too small to use. A Knoop-type indenter was made from a selected crystal of silicon carbide and another from boron carbide.[7] After many attempts in grinding and polishing these crystals, it was finally concluded that the inherent imperfections of the carbide samples militate against obtaining a geometrical perfection approaching that possible with the diamond. Apparently, there is a finite limit of sharpness that can be obtained in each material without incurring fragmentation. The result of imperfections in the indenter tip is of course a ragged, irregular impression and consequent inaccurate hardness value. The poor quality of these indenters precludes their use for any quantitative work.

The conclusion therefore results that, with respect to either material or geometry, no selection of indenter can be made at present which will be universally applicable for all test materials and temperature ranges. This problem would be a fruitful field for further research.[8]

## OPERATING CHARACTERISTICS AND LIMITATIONS

### *Loading:*

The major problem encountered in the construction of the tester described lay, as Bergh experienced, in achieving satisfactory piston operation. Any slight friction due to dust, rust, etc., in the piston-cylinder assembly itself or any drag imparted by the thermocouple wires led to erroneous results in two ways. First, if a steady friction were present which varied in amount between runs, the load-calibration factor was not constant and hence the true load became indeterminate. On the other hand, if a slight impediment were encountered momentarily during the upward motion of the piston and a corresponding increase made in the hydraulic pressure to overcome this, the result was an impact loading of the specimen. Thus, a fair-sized indentation would be made just in the application of the preload, which might be larger than the normal impression, particularly at small loads.

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[6] Thanks are due F. H. Horn of this laboratory for several SiC crystals, and L. H. Milligan of the Norton Co. for crystals of SiC, $B_4C$, and TiC.

[7] The author is indebted to Peter Vuille for his skill and persistence in this most difficult task. Mr. Vuille was also responsible for cutting one of the sapphire indenters previously mentioned.

[8] Since completion of this manuscript, limited success has been obtained with Vickers indenters of $TiB_2$. Crystals synthesized to date, however, are neither as large nor as perfect as would be preferred.

The motion of the piston must not only be very smooth, but also very slow, and the mass of the moving parts must be kept low, particularly if significant data are to be obtained for the very hard, brittle materials such as carbides, nonmetallics, or intermetallic compounds. In such materials, cracking is a frequent result of high loading speed, in addition to oversized impressions as described above. Bergsman (**14**) and Grishig (**15**) have made quantitative studies of the effect of loading speed. Bergsman's results on a hardened tool steel are reproduced in Fig. 5. It will be observed that the effect of loading speed becomes more pronounced with decrease in load. The machine described by Bergh operated at about 4 mm per min. Under optimum conditions, the tester described in this report could be run as slowly as 2 mm per min. As can be seen from Bergsman's plot, even lower speeds would be desirable but are not practical with manual operation. Studies are now under way for modifying the instrument described here for automatic operation.

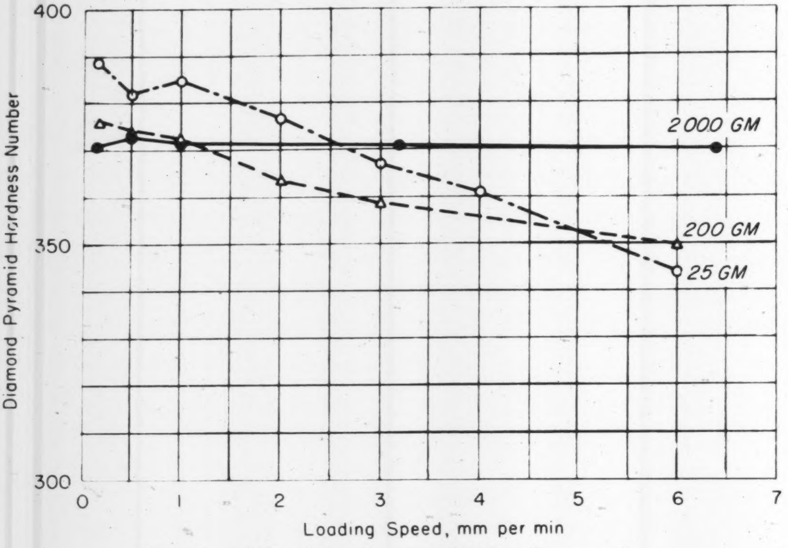
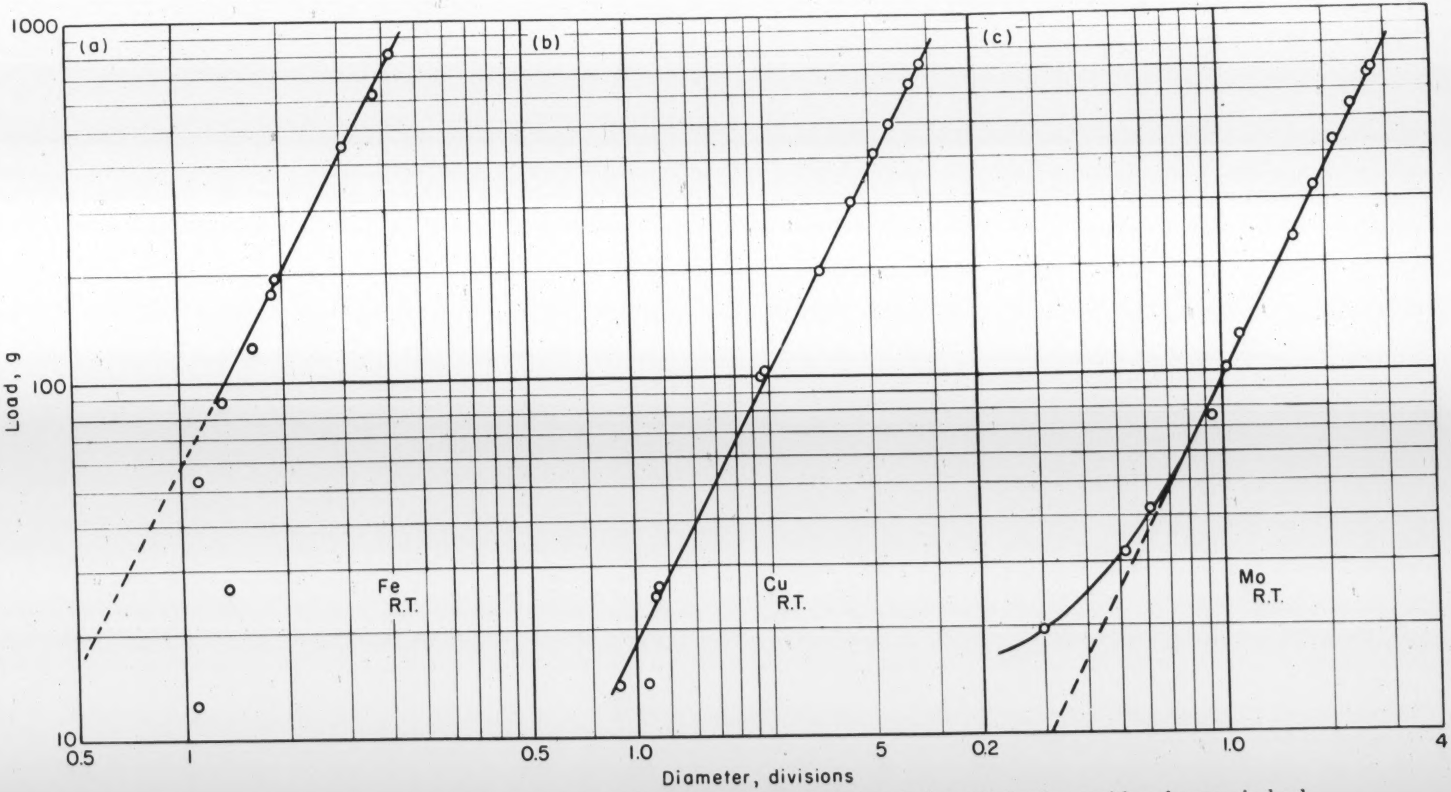


FIG. 5.—Effect of Loading Speed (After Bergsman).

To check on progress made in piston operation by cleaning the hydraulic system of loose particles, by improving surface finish, and by eliminating thermocouple drag, the following experiment was performed. Indentations were made in a specimen at room temperature with a variety of loads extending over the whole range of the instrument. Time under load was held constant at 15 sec. The size of each impression was then



(*a*) poor piston behavior. (*b*) satisfactory piston behavior. (*c*) indication of an increase in hardness with a decrease in load.

FIG. 6.—Loading Tests.

measured and the data plotted as load *versus* diameter on log-log coordinates. Since the hardness is by definition the mean stress over the indentation, the load should be proportional to the square of the lineal dimension of the indentation. Thus, the log-log plot of the data should result in a straight line of slope 2. A typical plot obtained early in the development of the tester is shown in Fig. 6(*a*). The deviation of points below the straight line for loads of less than 200 g is attributable to impact loading. It was considered that satisfactory piston operation had been obtained when results of the sort shown in Fig. 6(*b*) were achieved. Unfortunately, not all materials show an independence of hardness with load, as indicated by Fig. 6(*b*). More usually, particularly with hard, brittle materials, a significant increase in hardness with decreasing load is observed in the low-load range. This effect is illustrated by some data for molybdenum, shown in Fig. 6(*c*).

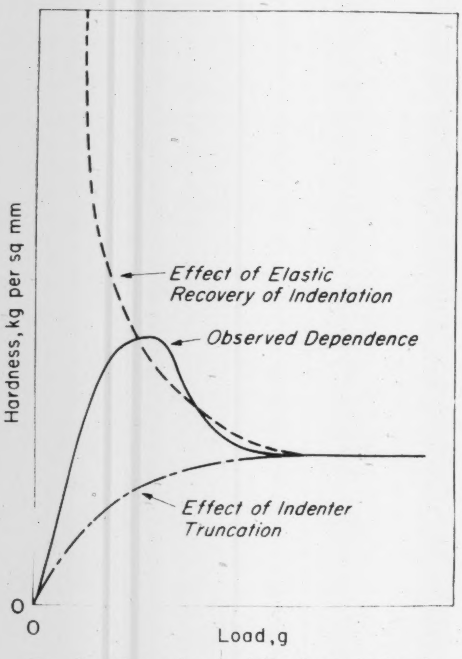


FIG. 7.—Dependence of Hardness on Load.

It has been established (**14,16**) that, at least for the range above about 100 g, this effect is due to elastic recovery of the indentation. The observed hardness is therefore expected to increase hyperbolically with decreasing load, as shown by the dashed curve of Fig. 7. The asymptote of this curve thus represents the highest load corresponding to completely elastic deformation. Tarasov and Thibault (**16**), as well as Bergsman (**14**), have shown that correction can be made for deviations of this type by adding a constant (determined by the best least squares fit to the straight line) to the observed length of the indentation. In certain cases, an even more complex dependence of hardness on load has been found (**17**); this is illustrated schematically by the full curve in Fig. 7. This curve has been interpreted by Grodzinski (**18**) as the result of the combination of two separate effects: elastic recovery and microform of the indenter. With respect to the latter, it has been reasoned (**18**) that, with decreasing loads, a point is eventually reached at which the contact area during indentation is determined not by the general geometry of the indenter but by its microscopic configuration at the tip. Since this tip configuration is generally a truncation or rounding of some sort, an apparent decrease in hardness with decreasing load is expected in the low-load range, as indicated by the dot-dash curve of Fig. 7. Since the magnitudes of both of these effects vary independently with material tested and with the indenter, the observed dependence of hardness on load may have a variety of shapes, of which that shown by the full curve is typical. On the other hand, the results of Campbell *et al* (**10**) showing decreasing hardness with decreasing load may be explained by assuming that, for their conditions, the truncation effect predominates. Inasmuch as the situation is not yet completely resolved, it is well to specify the load used when reporting data.

*Method of Operation:*

The apparatus can be operated in either of two ways. For survey purposes, it is often desirable to measure and compare the hardness values of a number of samples at the same temperature. This operation is readily accomplished by charging a number of specimens into the magazine and then bringing the test chamber to the desired temperature, where it is maintained by the controller. Test specimens are then brought one at a time from the magazine to the anvil, hardness impressions made, and the specimens finally pushed down the chute to the receptacle chamber. About twenty minutes is allowed for the specimen to come to furnace temperature. Test runs have been made as a function of time after charging, which demonstrated the adequacy of this warm-up period. On the other hand, it may be desirable to run isolated samples rather than a series, or to examine in detail the temperature-dependence of hardness of a single specimen. In this case, the test chamber is heated continuously and hardness impressions made at desired temperature intervals. By maintaining an accurate log of time, temperature, position, and loading data, a hardness-temperature plot can be readily derived from a single run. The temperature is raised at an approximately linear rate by manual adjustment of the voltage supplied the furnace coil. For heating rates up to at least 5 C per min, close checks were obtained between isothermal and continuous heating runs on the same sample. Figure 8 shows one such check run.

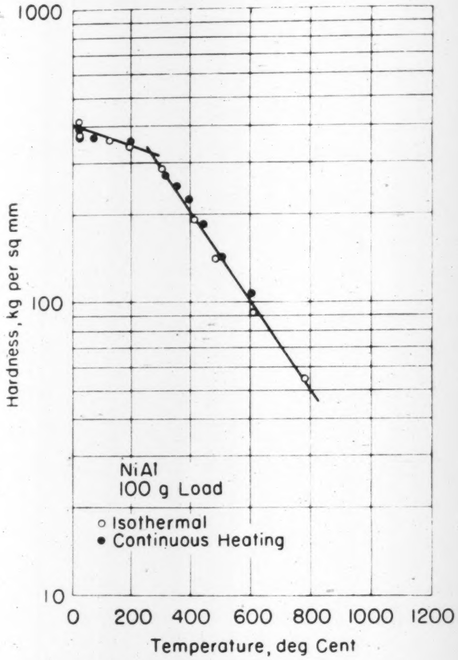


FIG. 8.—Hot-Hardness Test: Isothermal *versus* Continuous Heating.

*Measurement of Indentations:*

The accurate measurement of microhardness impressions is an exceedingly difficult, tedious, and exasperating task, especially when acutely elongated indentations of the Knoop or double-cone type are used. The resolving power of the optical system, calibration of the measuring device, and visual acuity of the observer are all critical factors. These subjects have been studied in considerable detail by Thibault and Nyquist (**19**) and by Bergsman (**14**). The present research adds little except confirmation of their results. Three optical systems have been used. For soft materials (<1000 diamond pyramid number), the optical system on the standard Tukon tester (6-mm objective, 0.65 numerical aperture (NA)) has proven satisfactory. For the hard, opaque materials, the 54×, 1.00 NA, oil immersion fluorite objective, and bright field illumination, recommended by Thibault and Nyquist, was used on a Bausch & Lomb research metallograph and proved to be the best. Slightly oblique lighting and use of a No. 78 blue Wratten filter gave a significant improvement in contrast. For the transparent or translucent hard materials such as sapphire, best results were obtained with a 58×, 0.85 NA dry objective, oblique light, and the No. 78 blue Wratten filter.

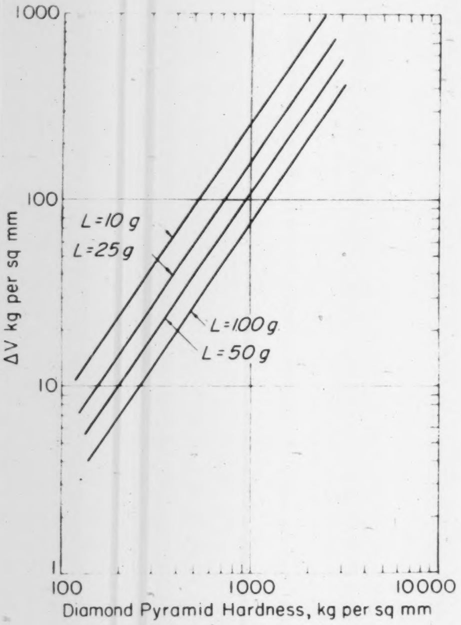


FIG. 9.—Effect of Measurement Error of 0.5μ on the Vickers Hardness.

Measurements of the size of impressions were made with a Bausch & Lomb filar eyepiece previously calibrated with a Reichert stage micrometer ruled with 100 lines per mm. Bergsman (**14**) believes that ordinary commercial stage micrometers are too crude for the degree of precision required in microhardness calibration studies. He therefore had an optical grating specially ruled for him with finer spacing, sharper rulings, and considerably higher accuracy. While this is undoubtedly a step in the right direction, it was felt in the present instance that other factors involved more appreciable errors, and hence purchase of a custom-made grating was not warranted. Bergsman also found that his calibration factor varied appreciably with magnitude of the distance measured. No such variation was found in the present work, although the grating used was less accurate than Bergsman's and fewer measurements were made (30 *versus* about 500).

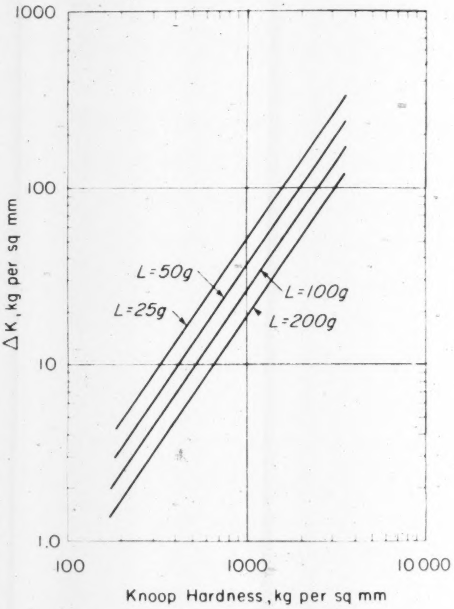


FIG. 10.—Effect of Measurement Error of 0.5μ on the Knoop Hardness.

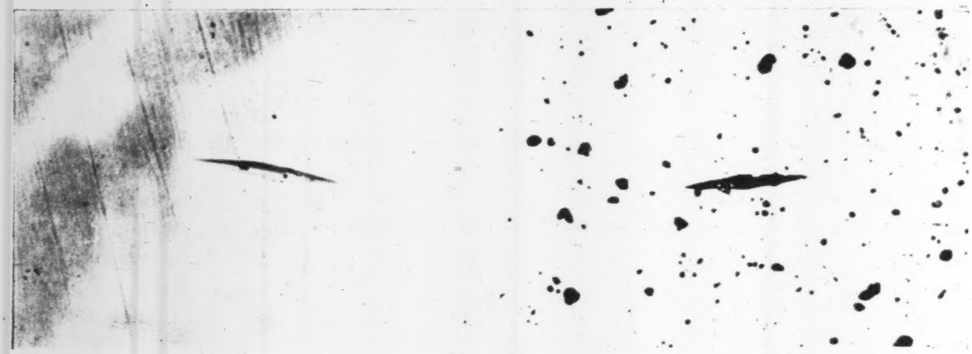
As mentioned above, the total error involved in the indentation measurement is made up of three component errors: the uncertainty in locating the precise limits of the indentation, the differences in visual acuity of observers, and the calibration error. Since these factors are essentially constant, the total relative error becomes greater with decreasing size of indentation (low load, high hardness). Bergsman, (**14**) Plaut and Thring (**20**) and Brown and Ineson (**21**) all agree that for the Vickers indentation, the total error is approximately $0.5\mu$. The latter authors have therefore calculated the hardness variation to be expected for various loads as a function of hardness level, assuming an increase of $0.5\mu$ in the diagonal length of a Vickers indentation. Their results are reproduced in Fig. 9. A similar plot for the Knoop indenter, derived by the present author, is shown in Fig. 10. It must again be emphasized, however, that the quantitative error in length in the measurement of a Knoop impression is much greater than for a Vickers impression of comparable size.

Certain other features of the metallography of microhardness impressions warrant comment. The importance of surface preparation of the test specimen has been well emphasized by previous investigators (**2,14,19**). In materials susceptible to work hardening by mechanical polishing, erroneous high hardness values are frequently obtained—the more so, the smaller the load used. Electropolishing is required in such instances. While it has been shown that for hard materials the problem of surface cold work by mechanical polishing is virtually nonexistent (**19**), another and more difficult aspect of the problem confronts us—the achievement of a smooth, solid surface, free of "pull out." Much has been written on special polishing techniques for hard materials; nevertheless, many materials exist for which no adequate techniques have yet been developed.

Spacing of the indentations must be controlled to avoid interference between indentations by cold work and cracking. Although no thorough study was made in this case, the author is in agreement with previous investigators (**19,22**) in recommending a minimum spacing of 1.5 times the length of indentation.

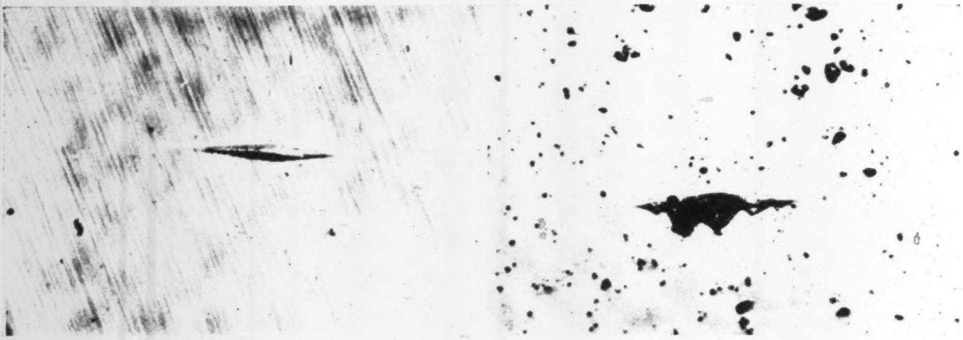
A further consideration is the possible effect of grain orientation on hardness. This problem may be handled in several ways. If single crystal specimens are available, they may be oriented relative to the indenter before measurements are made. The results are then reported, together with the crystal face investigated, and the orientation of the principal axis of the indenter on the crystal face. This practice was adopted by Winchell (**23**) in his studies of mineralogical specimens. On the other hand, possible effects of grain orientation may be averaged out. Thibault and Nyquist (**19**) made two to three indentations in each of 25 to 30 randomly oriented grains and then averaged the data for all satisfactory indentations. It may also be possible to use a load large enough that a comparable number of grains are encompassed by a single indentation. Data obtained on single crystals of unknown orientation or for cases where only a few grains are included in a single indentation must be regarded as of limited significance. Fortunately, many cubic materials show relatively small effects of orientation. When making hardness tests on a specimen with symmetrical indenters, any pronounced effects of orientation are immediately obvious in asymmetry of impressions. On the other hand, asymmetrical indenters such as the Knoop, which are more sensitive to the effects of orientation (**24**), do not reveal the presence of anisotropy by simple inspection of the impressions.

A similar problem exists with multiphase materials, which are not usually suitable for microhardness testing because of the difficulty of securing a statistically average sampling of the microstructure within the compass of a single indentation. Nor is it readily possible with this instrument, in contrast to



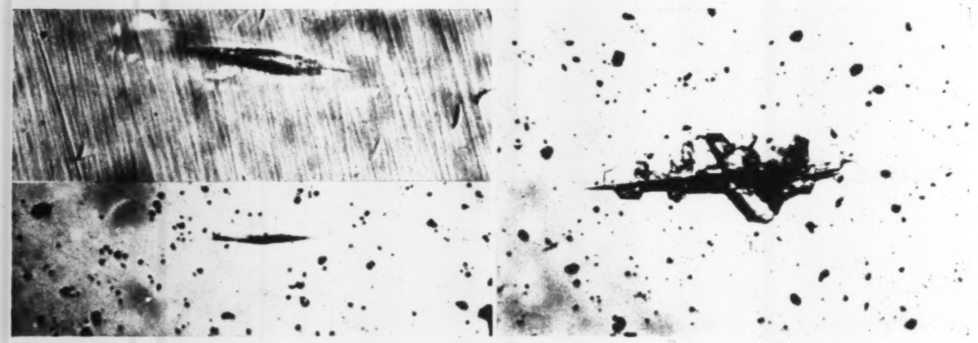
(*a*) Class 1, sapphire.

(*d*) Class 4, TiC.



(*b*) Class 2, sapphire.

(*e*) Class 5, TiC.



(*c*) Class 3, sapphire; Class 1 to 3 TiC.

(*f*) Class 6, TiC.

FIG. 11.—Illustrative Examples of the Classification Scheme for Degree of Cracking.

NOTE.—The metallographic preparation of these specimens is not considered adequate for quantitative work.

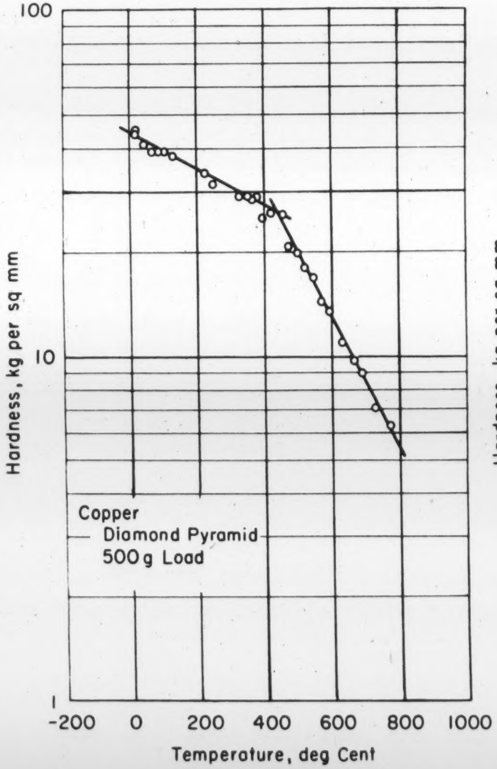


FIG. 12.—Hot-Hardness of OFHC Copper.

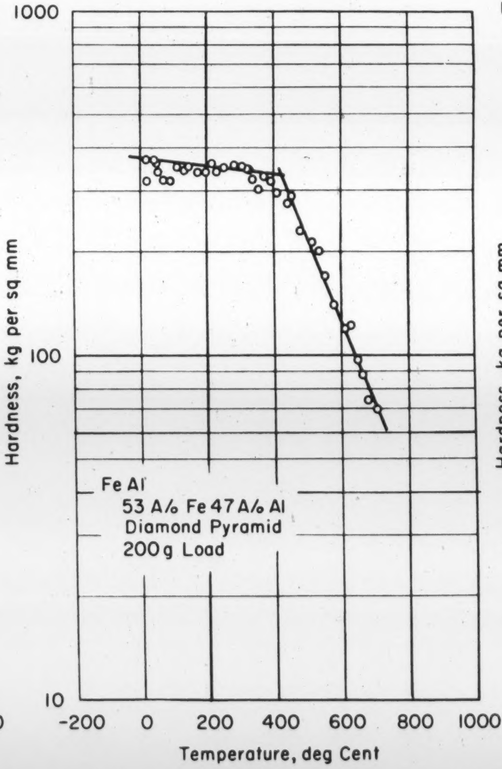


FIG. 13.—Hot-Hardness of an Iron Aluminide.

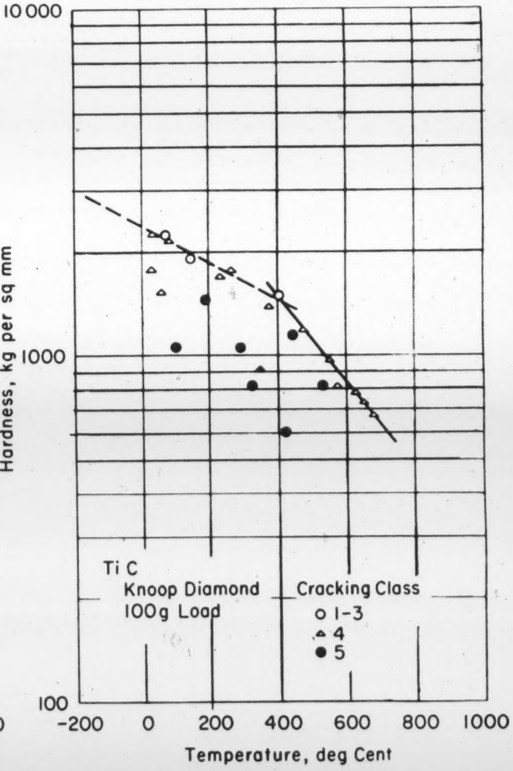


FIG. 14.—Hot-Hardness of Vacuum-Sintered TiC.

room-temperature microhardness testers, to measure the hardness of each phase independently by very small indentations at preselected sites. An effort in this direction might be attempted by making a "blind" traverse at constant temperature across a multiphase specimen and subsequently selecting for measurement only those impressions lying wholly within a given phase.

A final metallographic consideration in the microhardness testing of brittle materials is the effect of cracking on the hardness number. Thibault and Nyquist (**19**) studied this subject in some detail and arrived at a classification scheme defining six degrees of cracking. Classes 1 to 3 involve various degrees of subsurface cracking and hence can be distinguished only in transparent materials. Class 3 is defined as "no to slight" surface cracking, accompanied by some subsurface cracking. Classes 4, 5, and 6 represent progressively greater amounts of surface cracking. The differences in susceptibility of various substances have already been mentioned, as well as the effects of certain operating procedures. Thibault and Nyquist believe their Knoop numbers to be unaffected by cracking of classes 1 to 3. They would reject all data resulting from impressions of class 6 and view data from classes 4 and 5 with caution. In cases of doubt, they prefer to accept the results of the smaller indentations. The present author has made a similar study in a variety of hard materials ranging from intermetallic compounds through sapphire to sintered carbides. Impressions typical of the various cracking classes, taken from some of the author's early studies, are shown in Fig. 11. The distribution of hardness impressions among the cracking classes shifts toward the lower crack-free classes as the temperature is increased. In addition, the points at the upper edge of the envelope of data tend to be predominantly in classes 1 to 3. These findings are discussed further below.

TABLE II.—TYPICAL SCATTER IN THE ROOM TEMPERATURE MICROHARDNESS OF HARD MATERIALS (**19**).

| | $K_{100}$ Mean | Range, per cent of Mean |
|---|---|---|
| $B_4C$ | 2800 | 9.6 |
| SiC | 2480 | 20.6 |
| TiC | 2470 | 10.9 |
| Synthetic sapphire | 1950 | 21.6 |
| WC | 1880 | 30.4 |
| Topaz | 1340 | 19.4 |
| Quartz | 820 | 14.6 |
| Hardened tool steel | 740 | 4.1 |

*Typical Results, Analysis, and Comparison:*

Figures 12, 13, and 14 show some of the results obtained in this machine for several different types of material—a soft, ductile metal; an intermetallic compound of moderate hardness and limited ductility; and a carbide of extreme hardness and brittleness.[9]

The semilogarithmic method of plotting has been used, since it has been demonstrated (**25**) that this results, for virtually every case, in a two-segmented linear plot, which is of great convenience in analysis of data. As may be noted, the scatter of the data increases with increasing hardness. However, the scatter is also affected markedly by the ductility of the material and to a lesser extent by other factors, for example, load and contrast of impression. Table II, based upon data taken by Thibault and Nyquist (**19**), shows the magnitude of scatter obtained by very careful workers at constant load and under optimum optical conditions for a variety of hard materials. Their mean values were obtained by averaging 100 or more *selected* impressions. These authors estimated that 15 to 20 man-hours were required to obtain each of the averages shown. It is obviously impractical to attempt to measure hot-hardness with such thoroughness. It appears better, for those materials susceptible to wide scatter, to take a reasonable number of points over the temperature range of interest, say 30 to 40 points for an 800 C range; to plot all points for which visually acceptable impressions were obtained; and then to represent the data by the best linear plot approximating the upper envelope of the whole scatter band. This procedure is in accord with the view taken by Winchell (**23**) in analyzing the results of room-temperature microhardness tests on brittle materials.

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[9] The data shown for the Knoop indentations in TiC were calculated on the basis of the contact area rather than the projected area of the indentation as called for in the Knoop formula in order to place all hardness data obtained in this study on a more equivalent basis. True Knoop numbers are about 10 per cent higher than hardness numbers calculated from contact areas.

TABLE III.—COMPARATIVE DATA ON THE MICRO-HARDNESS OF VARIOUS MATERIALS.

| Material | Hardness Data, kg per sq mm | | | | |
|---|---|---|---|---|---|
| | Temperature, deg Cent | This Instrument | Standard Tukon$^{a}$ | Literature | Literature Reference |
| TiC | room temperature | 2430$^{b}$ | 2470 | 2350 to 3200$^{c}$ | **19, 26, 27** |
| Sapphire | room temperature | 2180$^{b}$ | 2100 | 950 to 2250$^{c}$ | **18, 19, 23, 28** |
| Quartz | room temperature | 690$^{b}$ | 742 | 650 to 1230$^{c}$ | **11, 18, 19, 23, 29, 30** |
| Molybdenum | room temperature | 150$^{d}$ | 147 | 168$^{e}$ | **31** |
| | 500 | 72 | | 83 | |
| | 800 | 64 | | 72 | |
| Copper OFHC | room temperature | 44$^{f}$ | 45 | 49$^{g}$ | **32, 33** |
| | 300 | 31 | | 37 | |
| | 500 | 20 | | 16 | |
| | 800 | 5.4 | | 4.5 | |

[a] Same specimen, load, and indenter type as for data from this instrument.

[b] 100-g load, Knoop diamond indenter.

[c] Various loads and indenter types; see references. Spread in hardness values is affected by specimen orientation as well.

[d] 400-g load, Vickers diamond indenter

[e] 10-kg load, Vickers diamond indenter.

[f] 500-g load, Vickers diamond indenter.

[g] 10 to 150-kg load, 120-deg cone indenter (diamond <600 C, sapphire> 600 C).

It is also of interest to compare data obtained with this machine with those reported by other investigators working with other machines. Table III shows such a tabulation. While better correspondence might be desired, the checks are considered to be rather good, particularly since tests were not made on identical specimens. The agreement also looks close in the light of the results of Brown and Ineson (**21**), who measured the microhardness of eight metallic materials with 13 different sets of apparatus. The wide variation in results which they obtained is illustrated in Fig. 15. Inasmuch as certain unavoidable variations in load were present between various machines which they tested, hardness numbers were calculated for purposes of this comparison on the basis of a constant length of indentation (10$\mu$). The variability between machines is very great, but, in fairness, it must be pointed out that the samples used by Brown and Ineson were not very homogeneous except for Nos. 1, 2, and 3. This condition

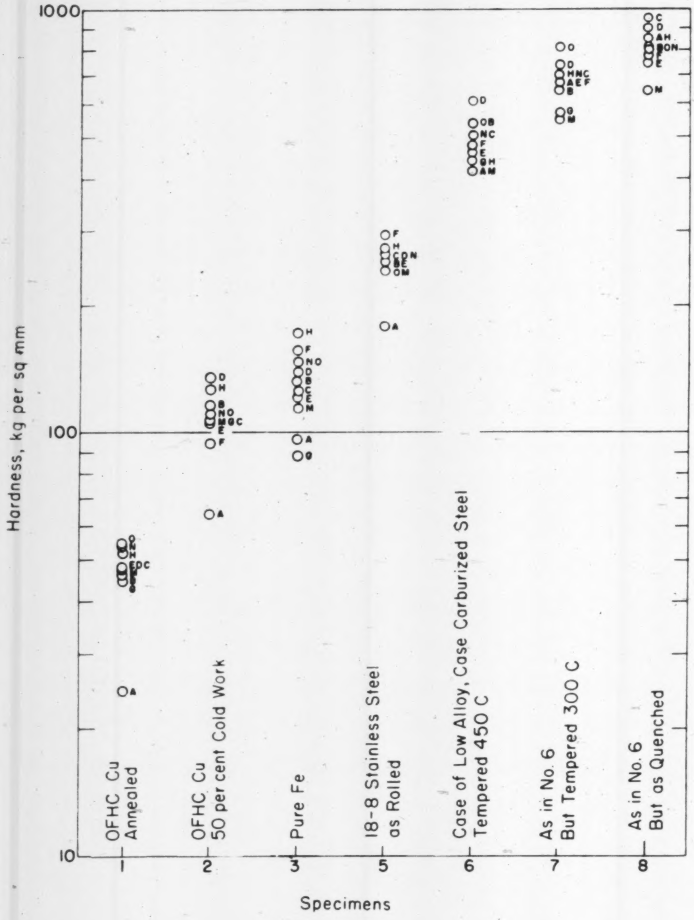

FIG. 15.—Microhardness of Identical Specimens as Measured by Different Instruments.

is undoubtedly also responsible for much of the scatter found by them in a single sample tested with one machine. In view of their findings and the present author's experience, the recommendations of Brown and Ineson for the significance level of hardness difference shown in Table IV are thought to be well founded. On the other hand, it must be pointed out that it has been found possible for different workers to obtain excellent agreement in hardness values when

TABLE IV.—DEFINABLE DIFFERENCE IN HARDNESS.

| Range, VPN | Difference Definable VPN |
|---|---|
| 0 to 100 | 20 |
| 100 to 400 | 50 |
| 400 to 1000 | 100 |
| >1000 | 200[a] |

[a] Specification by present author.

testing samples of the same material with instruments of identical design under very carefully controlled conditions (**34**). Table V, taken from reference **34**, shows one such example.

TABLE V.—EXAMPLE OF AGREEMENT BETWEEN WORKERS.

| Material | Mean Knoop$_{100g}$ Values (**34**) | |
|---|---|---|
| | Kenna-metal | Norton Co. |
| Boron carbide | 2808 | 2800 |
| Tungsten titanium carbide | 2160 | 2190 |
| Cemented carbide, grade K6 | 1788 | 1800 |
| Cemented carbide, grade KM | 1487 | 1500 |

## APPLICATIONS

The primary purpose for which the instrument was built was to measure the temperature-dependence of hardness of brittle (single-phase) materials—ionic crystals, intermetallic compounds, carbides, borides, etc. Certain portions of this work have already been reported (**35,36,37**). Some work has also been undertaken, however, for other individuals with quite different objectives. A few examples may prove informative.

It was desired to determine the potentialities of mild steel as a sheath material and to establish the optimum working temperatures for the warm-rolling of high-purity chromium. Hardness curves run on samples of both materials indicated that hardness values become comparable in the range 600 to 650 C, and hence this should be a satisfactory temperature for sheath rolling (**38**).

In another instance, the investigator wished to know the effect of operating time and temperature in a liquid-sodium environment on the hardness of a high-temperature alloy at both room and elevated temperatures. The application was for a bearing and structural member in a mechanism associated with an atomic pile. Contrary to expectation, the alloy showed a slight increase in hardness at 500 C over the room-temperature value before exposure to sodium, and only a slight softening with time in liquid sodium at 500 C (**39**). The hardening is evidently due to an aging reaction.

Efficient surveys of new compositions of high-temperature alloys are also feasible. From hot-hardness data on samples at selected temperature levels, promising compositions can be separated for further study with more refined techniques.

The hot-hardness test is also adaptable to many other types of investigations. It may be used, for example, in studying the kinetics of many metallurgical processes, for example, recovery, recrystallization, polygonization (**40**), diffusion (**41,42**), phase transformation (**43,44**), etc. Garofalo *et al.* (**45**) have found it useful in studying the strain-aging phenomenon in steels. It has even been proposed (**46**) as a tool for phase-diagram determination. Onitsch (**47**) has used hot-hardness in studying the sintering process.

### *Acknowledgment:*

The author is indebted to many people for assistance and advice during the course of this work. Special thanks are due Edwin Matthews and Charles Arnold, who not only were responsible for the construction of the instrument, but also made major contributions toward its design. A. Casagrande made the original adaptation of the Bergh design. The assistance of Eugene Paquette during the early stages of the work is gratefully acknowledged. Certain of the hardness data illustrated in this report were obtained by Arthur Peat, whose contribution is also greatly appreciated.

## REFERENCES

**(1)** F. P. Bens, "Hardness Testing of Metals and Alloys at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 38, p. 505 (1947).

**(2)** F. Garofalo, P. R. Malenock and G. V. Smith, "Hardness of Various Steels at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 45, p. 377 (1953).

**(3)** I. L. Mirkin and D. E. Livshits, "A Method of Hardness Testing at High Temperatures," *Zavodskaya Laboratoria*, No. 9, p. 1080 (1949).

**(4)** R. F. Domagala and W. R. Johnson, "Simple Hot Hardness Tester," *Metal Progress*, Vol. 60, p. 72 (1951).

**(5)** J. Engl and G. Heidtkamp, "Temperature Dependence of the Cone Hardness of Metals, I," *Zeitschrift für Physik*, Vol. 95, p. 30 (1935).

**(6)** A. Brenner, "Microhardness Tester for Metals at Elevated Temperatures," *Journal of Research*, Nat. Bureau Standards, Vol. 46, p. 126 (1951).

**(7)** P. S. Bergh, "High Temperature Microhardness Tester," *Technical Report No. M-5*, Silver Springs Lab., The Kellex Corp., Dec. 1948.

**(7a)** E. S. Berkovich, "Triangular Diamond Pyramid for Studying Micro Indentation Hardness," *Zavodskaya Laboratoria*, Vol. 16, p. 345 (1950); see also abstract translation *Industrial Diamond Review*, Vol. 11, p. 129 (1951).

**(8)** P. Grodzinski, "Improvements in Hardness Testing, in Particular in Microhardness Testing," *British Patent 651,800.*

**(9)** P. Grodzinski, "Damaged Impressions in Microhardness Testing," *Research*, (London), Vol. 4, p. 140 (1951).

**(10)** R. F. Campbell, Q. Henderson and J. R. Donleavy, "A New Design of Microhardness Tester and Some Factors Affecting the Diamond Pyramid Hardness Number at Light Loads," *Transactions*, Am. Soc. Metals, Vol. 40, p. 954 (1948).

**(11)** F. Knoop, C. G. Peters and W. B. Emerson, "Sensitive Pyramidal-Diamond Tool for Indentation Measurements," *Journal of Research*, Nat. Bureau Standards, Vol. 23, p. 39 (1939).

**(12)** H. D. Leighly, Jr. and H. L. Walker, "Titanium Sticks to Indenter," *Metal Progress*, Vol. 62, p. 109 (1952).

**(13)** R. W. Hanzel, "More on Titanium Sticking to Indenter," *Metal Progress*, Vol. 64, p. 112 (1953).

**(14)** E. B. Bergsman, "Improvements in Microhardness Testing," ASTM BULLETIN, No. 176, Sept., 1951, p. 37 (TP175).

**(15)** M. R. Girshig, "A Novel Apparatus for Microhardness, The 'Microscelerometer L.C.,'" *Revue de Métallurgie*, (Mémoires), Vol. 43, p. 95 (1946).

**(16)** L. P. Tarasov and N. W. Thibault, "Determination of Knoop Hardness Numbers Independent of Load," *Transactions*, Am. Soc. Metals, Vol. 37, p. 331 (1946).

**(17)** H. Meincke, "The Determination of the Maximum Hardness by the Ball-Pressing Procedure," *Zeitschrift für Metallkunde*, Vol. 34, p. 289 (1942).

**(18)** P. Grodzinski, "Micro-Indentation Hardness, a New Definition," *Industrial Diamond Revue*, Vol. 12, p. 209 (1952).

**(19)** N. W. Thibault and H. L. Nyquist, "The Measured Knoop Hardness of Hard Substances and Factors Affecting its Determination," *Transactions*, Am. Soc. Metals, Vol. 38, p. 271 (1947).

**(20)** H. C. Plaut and M. W. Thring, "A Note on Microhardness Testing," *Report No. MG/E/5/46*, B1SRA, quoted by Brown and Ineson (**21**).

**(21)** A. R. C. Brown and E. Ineson, "Experimental Survey of Low-Load Hardness Testing Instruments," *Journal of the Iron and Steel Institute*, Vol. 169, p. 376 (1951).

**(22)** D. R. Tate, "A Comparison of Microhardness Indentation Tests," *Transactions*, Am. Soc. Metals, Vol. 35, p. 374 (1945).

**(23)** H. Winchell, "The Knoop Microhardness Tester as a Mineralogical Tool," *Am. Mineralogist*, Vol. 30, p. 583 (1945).

**(24)** H. Meincke, "Hardness Testing with Asymmetrical Indenters," *Metalloberfläche*, Vol. 5A, p. 17 (1951).

**(25)** J. H. Westbrook, "Temperature Dependence of the Hardness of Pure Metals," *Transactions*, Am. Soc. Metals, Vol. 45, p. 221 (1953).

**(26)** R. Kieffer and F. Kölbl, "Tungsten Carbide Free Hard Metals," *Powder Metallurgy Bulletin*, Vol. 4, p. 4 (1949).

**(27)** J. Hinnüber, "Properties of Carbide Products," *Zeitschrift der Vereines deutscher Ingenieure*, Vol. 92, p. 111 (1950).

(28) C. Attinger, "Orientation and Hardness of Synthetic Corundum," *Bulletin Soc. Suisse de Chronométrie,* June, 1951; See extract in *Industrial Diamond Revue,* Vol. 12, p. 136 (1952).

(29) P. Mitsche and E. M. Onitsch, *Mikroskopie,* Vol. 3, p. 257 (1949).

(30) C. G. Peters and F. Knoop, "Metals in Thin Layers — Their Microhardness," *Metals and Alloys,* Vol. 13, p. 292 (1940).

(31) J. L. Ham, "An Introduction to Arc Cast Molybdenum and its Alloys," *Transactions,* Am. Soc. Metals, Vol. 73, p. 723 (1951).

(32) J. Engl and J. Folmer, "Temperature Dependence of the Cone Hardness of Metals, II," *Zeitschrift für Physik,* Vol. 98, p. 702 (1936).

(33) J. Engl and J. Katz, "Temperature Dependence of the Cone Hardness of Metals, III," *Zeitschrift für Physik,* Vol. 106, p. 1 (1937).

(34) E. E. Wicker and J. C. Redmond, discussion of paper by Thibault and Nyquist (**19**).

(35) J. H. Westbrook, "Temperature Dependence of Hardness of the Equiatomic Iron Group Aluminides," *Journal,* Electrochemical Soc., Vol. 103, p. 54 (1956).

(36) J. H. Westbrook, "Note on the Temperature Dependence of Hardness of the Transition Metal Monocarbides," *Acta Metallurgia,* Vol. 3, p. 104 (1955).

(37) J. H. Westbrook, "Defect Structure and the Temperature Dependence of Hardness of an Intermetallic Compound," *Journal,* Electrochemical Soc., Vol. 104, p. 359 (1957).

(38) Private communication, J. H. Westbrook to W. H. Smith, GE Research Laboratory.

(39) Private communication, J. H. Westbrook to E. L. Baldwin, Knolls Atomic Power Lab.

(40) K. A. Osipov, S. G. Fedotov and M. G. Lozinsky, "On a New Mechanism of Plasticity of Metallic Solid Solutions," *Doklady Akademii Nauk.* Soyuza Sovetskikh Sotsialisticheskikh Respublik, Vol. 89, p. 57 (1953); *Translation 19,* Nat. Science Foundation.

(41) H. Bückle, "Measurement of Diffusion with the Microhardness Tester," *Zeitschrift für Metallkunde,* Vol. 34, p. 130 (1942).

(42) U. Zwicker, "Measurement of Diffusion Layers in Iron by Means of Microhardness," *Metalloberfläche,* Series A, Vol. 6, June, 1952, p. A81.

(43) M. Casadio, "On the Determination of Temperature - Time - Transformation Curves," *Metallurgica italiana,* Vol. 45, p. 163 (1953).

(44) W. Chubb, "Contribution of Crystal Structure to the Hardness of Metals," *Transactions,* Am. Inst. of Metals, Vol. 203, p. 189 (1955).

(45) F. Garofalo, D. C. Marsden and G. V. Smith, "The Temperature Dependence of the Hardness of Pure Iron and Various Ferritic Steels," *Transactions,* Am. Soc. Metals, Vol. 49 (1957).

(46) V. P. Shishokin, "The Hardness and Fluidity of Metals at Different Temperatures," *Zeitschrift für anorganische Chemie,* Vol. 189, p. 263 (1930).

(47) E. M. Onitsch-Modl, "Micro-Hardness as an Expedient for Tests of Sintering Processes in Complex Systems," *Plansee Proceedings,* Metallwerk Plansee, GmbH, p. 175 (1952).

# DISCUSSION

Mr. Samuel J. Rosenberg.[1]—In the lecture presented by Mr. Herzig,[2] he presented data on hardness values taken at, as I recall, about 3000 F. Does the author know of the techniques that were used by Mr. Herzig?

Mr. J. H. Westbrook (*author*).—Yes, I do. I believe that work is about to be reported in the Am. Soc. Metals, *Transactions* for this year. At least I have seen such a paper from their laboratory and I presume it is the same equipment.

This is a very nice piece of work they have done. Their equipment, however, would not have solved the problems which we face in our work on hard, brittle compounds in that their machine is a high-load machine rather than a low-load machine, their indentor material is sapphire, and their range of experimental materials is relatively narrow, namley, molybdenum and tungsten-base alloys. With these last two conditions, they do not get into quite so many difficulties due to this specimen-indentor reaction. The other features of their equipment are not too dissimilar to those described here; that is, it too is an indentation type hardness test with means for positioning the specimen beneath the indentor, for measuring temperatures, for varying loads, etc.

Mr. M. Semchyshen.[3]—In the hot-hardness tester at Climax Molybdenum Co. laboratory, we use purified argon as the ambient atmosphere. In determining the hardness of reactive metals at temperatures up to 3000 F, we note that the surface of the specimens react with the atmosphere, thus giving rise to inaccurate hardness values. In the hardness apparatus described by the author, I note that the tests are conducted in vacuum. Has any effect of surface reaction with the ambient atmosphere been noted, especially when reactive metals are being investigated?

Mr. Westbrook.—Our vacuum, I would say, was fairly good for this sort of work. We maintained a vacuum of $\frac{2}{10}\,\mu$ or better over the whole temperature range, and except for some work we did with chromium some time back, we experienced no difficulty, that is, we could not see any difference between the hardness values we got when we varied the atmosphere. We have worked with inert gases also, but prefer vacuum except for cases where the sample contains a volatile element.

Mr. David E. Driscoll.[4]—The author gave a very clear analysis of his problems with the penetrator but ignored the equally difficult problem of the bond between the penetrator and loading rod. Some penetrators will withstand high temperatures but do not perform satisfactorily because the bond used between penetrator and loading rod flows at high temperature. I wonder what he has done to overcome this difficulty.

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[1] Metallurgist, National Bureau of Standards, Washington, D. C.

[2] Alvin J. Herzig, "A Perspective of Molybdenum-Base Alloys," Gillett Memorial Lecture, Am. Soc. Testing Mats., 60th Annual Meeting, June 1957. (Issued as separate ASTM publication.)

[3] Metallurgical Supervisor, Climax Molybdenum Company of Michigan, Detroit, Mich.

[4] Chief, Mechanical Metallurgy Branch, Watertown Arsenal, Watertown, Mass.

MR. WESTBROOK.—You are quite right. There is a very real problem here which I neglected to treat adequately. This, incidentally, aside from the graphitization problem, is another thing which presently puts the upper limit on the use of diamond indentors. The currently employed bonding materials soften badly or volatilize in vacua of the order we are working with here at just about the graphitization temperature.

I have not attempted a direct solution in my own work because there is so little temperature range left which is accessible because of the graphitization. However, it is possible to bond diamond directly to steel, nickel or various other substrates using the titanium hydride techniques which have been extensively employed in metal-ceramic sealing operations.

In the case of other indentor materials, we have had some success using various types of mechanical mounting. In the case of sapphire this is particularly easy because of the ready availability of sapphire in rod form. For instance, it is possible to mount the sapphire in a molybdenum head with a ground conical shoulder of the sapphire rod resting on a corresponding seat in the metal head. The tip of the rod, which is allowed to project through a hole in the molybdenum head, is then ground and polished to the desired geometry. We have had a considerable degree of success in this way.

I think this does not by any means exhaust the things that one might do, but you are quite right in pointing out that this is another important problem.

MR. THOMAS A. HUNTER[5] (*by letter*).—One small detail in the method of operation is not quite clear to me. I should like to know what happens to the specimens after they have been pushed down the chute to the receptacle chamber. Is the indentation measured while the specimen is hot? If so, how? If measured after the specimen has cooled, what sort of corrections are made to account for the dimensional changes caused by thermal expansion or contraction?

MR. WESTBROOK (*author's closure*).—As pointed out in the text of the paper the indentations are measured after cooling to room temperature. No correction is made for the contraction from the test temperature to room temperature since neglect of this error is less than the uncertainties in the final result introduced by other factors. This particular point has been discussed more fully by Bens (**1**).[6]

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[5] Manager, Ampatco Laboratories Corp., Cleveland, Ohio.

[6] The boldface number in parentheses refer to the references appended to the paper, see p. 894.

# AN EXPERIMENTAL STUDY OF COMPRESSIVE-CREEP BEHAVIOR AT ELEVATED TEMPERATURES*

By R. L. Carlson[1] and E. G. Bodine[2]

## Synopsis

Strain-*versus*-time curves for specimens of the aluminum alloy 2024-T4 tested in uniform compression exhibited decreasing creep rates during the initial part of the tests, and increasing creep rates during the final stage. Since the cross-sectional area increases with time in compression, the results cannot be attributed to a "necking" of the specimens. Metallographic examination of the specimens did not reveal any evidence of "internal" cracking. A review of the aging characteristics of this alloy for the test temperatures, however, indicates that the accelerated creep rate is probably associated with the loss in strength that accompanies overaging.

The results of creep-buckling tests on short columns of aluminum alloy 2024-T4 are presented. The decrease in capacity between 350 and 450 F is shown to be quite marked. Capacity estimates based on Shanley's time-dependent tangent-modulus method reflect the loss in capacity, but the values obtained are conservative for the column test data.

In recent years interest in the effect of creep behavior on the load-carrying capacities of structural elements has of necessity increased markedly. As a result, a considerable amount of research has been devoted to problems associated with elevated-temperature operation, and the literature in this general area has expanded rapidly (**1**).[3]

One of the objectives of the investigation summarized by this paper was to obtain uniaxial creep data for the stress range that would be encountered in the compressive loading of short columns. Although creep data are available for the relatively high stresses of interest, they are predominately for tensile loading.

A second objective was to study the characteristics involved in the creep buckling of "short"[4] columns. Although a number of theoretical treatments of column-creep buckling have been reported, relatively few experimental data are available (**2**, **3**).

The final objective of the investiga-

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Assistant Division Chief, Battelle Memorial Inst., Columbus, Ohio.

[2] Research Engineer, Battelle Memorial Inst., Columbus, Ohio.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 904.

[4] Perfectly straight, axially loaded "short" columns buckle inelastically; "slender" columns buckle elastically.

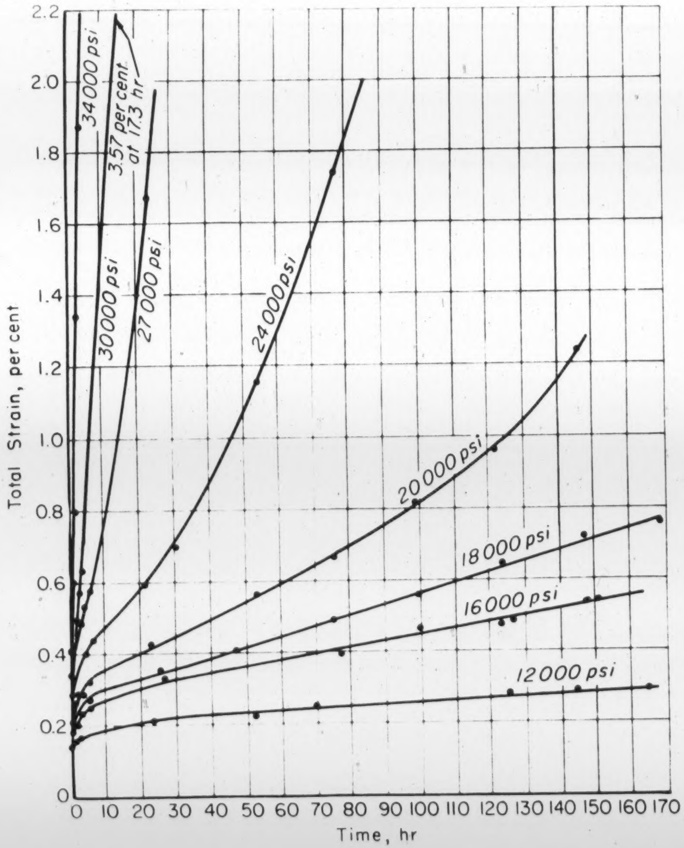

FIG. 1.—Total Strain-*versus*-Time Curves for As-Received 2024-T4 Aluminum Alloy Tested in Compression at 450 F.

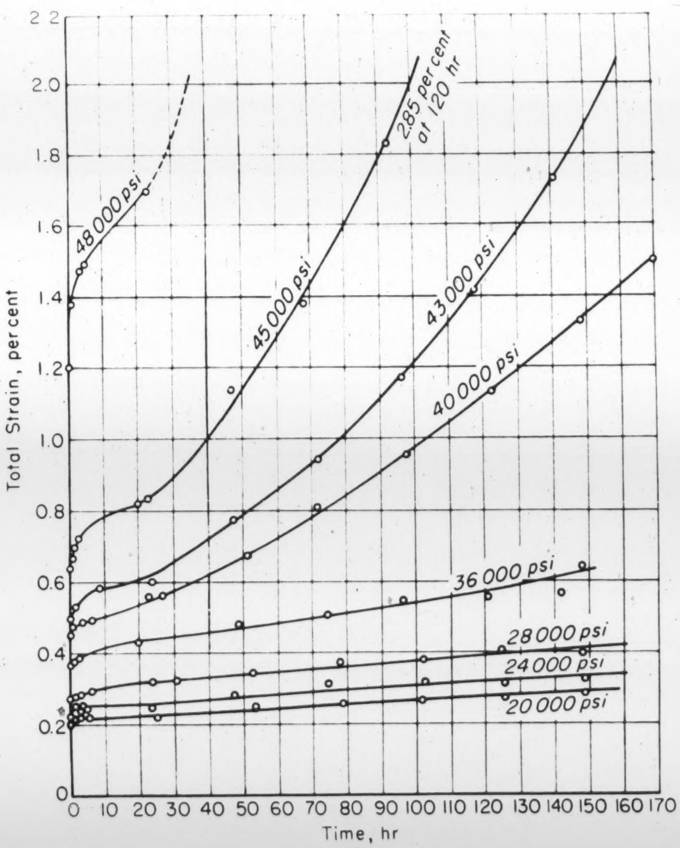

FIG. 2.—Total Strain-*versus*-Time Curves for As-Received 2024-T4 Aluminum Alloy Tested in Compression at 350 F.

tion was to correlate the data for uniform compressive loading with the data for columns.

The material used for this investigation was 2024-T4 aluminum alloy. All specimens were fabricated from $\frac{5}{8}$-in. rod stock which was obtained from a single heat. The test temperatures were 350 and 450 F. Details of the test equipment used are described elsewhere (3), so they will not be presented here.

## COMPRESSIVE-CREEP TESTS

Since the test material is unstable at the test temperatures used, care had to be exercised in reproducing the same heating time prior to testing. This time was 1 hr.

Test specimens were right-circular cylinders with a diameter of 0.312 in. and lengths of 1.25 and 0.87 in. The shorter length was used at the highest stress levels tested. This became necessary when it was observed that for the higher values of stress, creep buckling occurred and satisfactory creep data could not be obtained.

The results for the compressive-creep tests are plotted in Figs. 1 and 2. A particularly interesting feature of the curves is that after an initial period during which the creep rate steadily decreases, an inflection point occurs. Beyond that point the creep rate steadily increases. In view of the steadily increasing cross-sectional area that would be present in a compression-creep test, the accelerated creep rate may at first be difficult to understand. It was thought at first that alignment may have had some influence on this behavior. This was checked for all of the tests conducted, and it was found that in nearly all instances alignment was good within the strain ranges shown.

Sully, Cole, and Willoughby (4) observed alternately accelerating and decelerating creep rates in nickel-chromium alloys tested in compression at 800 to 900 C during what they defined as the tertiary stage. They suggested that the observed behavior may have been due to alternate hardening associated with the blocking of dislocations, and softening due to recovery processes akin to the onset of recrystallization. Greenwood and Warner (5), as well as others, had previously shown that recrystallization occurring during creep can apparently produce an increased rate of flow. Although the strain associated with the creep may be expected to lower the temperature at which recrystallization could occur, the test temperatures used in the present investigation, particularly 350 F, seem somewhat low for such effects. Further, metallographic examination indicated no evidence in support of such an explanation.

Machlin (6) has proposed a creep-rupture theory which predicts that rupture in tensile loading is preceded by the growth of pre-existent voids by vacancy-condensation mechanism. He also suggests that it should be possible to form visible voids by such a mechanism in specimens undergoing compressive creep.

Chen and Machlin (7) found numerous intercrystalline voids in creep specimens of alpha brass that had been compressed about 9 per cent at 1000 F. They suggested that the voids were nucleated by grain-boundary slip and that void growth occurred by vacancy condensation (6). In view of the fact that void formation was observed only for "high-temperature"[5] creep conditions, it should probably not be expected that the behavior observed in Figs. 1 and 2 for relatively low temperatures can be attributed to a deterioration associated with void formation. Nevertheless, several specimens were sectioned and examined. No voids were observed.

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[5] "High-temperature" creep conditions as used here refer to creep conditions for which the contribution of grain-boundary deformation is significant.

A more promising clue to the mechanism responsible for the observed behavior was found in the work of Finlay and Hibbard (8) who observed what they called an "intermittent creep rate" for a 4 per cent aluminum-copper alloy. In tests that they conducted, it was observed that the presence of the "intermittent creep rate" could be associated with precipitation phenomena.

If precipitation occurring during a test is responsible for the form of the curves of Figs. 1 and 2, one would expect that precipitation resulted in hardening of the test material up to times of approximately 10 to 20 hr at 350 F and about 5 hr at 450 F. Figure 3, which is a plot of yield strength *versus* aging time for several test temperatures, tends to support this thesis. The curves of Fig. 3 were obtained by Dix (9) for 2024-T4 flat sheet. Although these curves were obtained for the material in a different form, the aging characteristics should be expected to be similar to those of the test material used in this investigation. As can be seen, hardness increases steadily up to approximately 16 hr for a temperature of 350 F. Thereafter, the hardness increases only slightly to 30 hr. Unfortunately, the curves were not extended beyond 32 hr. The curves in Fig. 2, however, would tend to suggest that no further increase in hardness would be experienced. There might, in fact, be a slight decrease after 32 hr. These data, then, tend to support the suggestion that the exhibited resistance to creep is associated with aging phenomena.

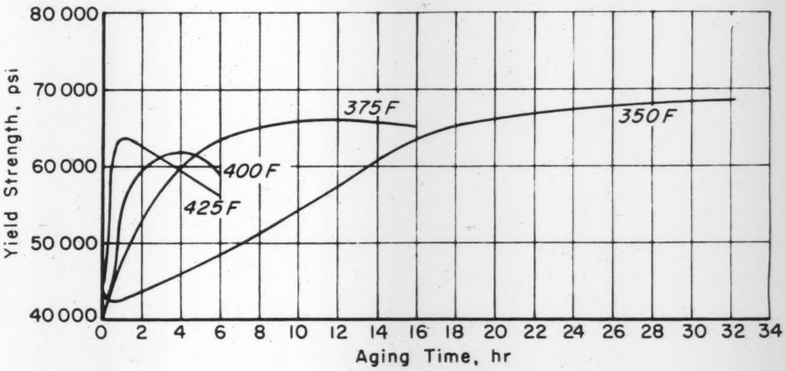

Fig. 3.—Artificial Aging Curves for 2024-T4 Flat Sheet (After Dix (9)).

Work by Underwood (10) has indicated that the precipitation that occurs during creep can be expected to be influenced by the strain that is occurring. There is, in essence, an interaction effect. Since the aging curves of Fig. 3 were obtained for specimens in an unstressed state, their form with respect to obtaining times for creep-response changes should, therefore, be used only as a qualitative guide.

Unfortunately, the plot of Fig. 3 does not include an aging curve for the temperature of 450 F. The forms of those presented, however, do suggest what may be expected. The curves given would tend to indicate that an increase in hardness would occur, but that hardness would not remain at a high level much

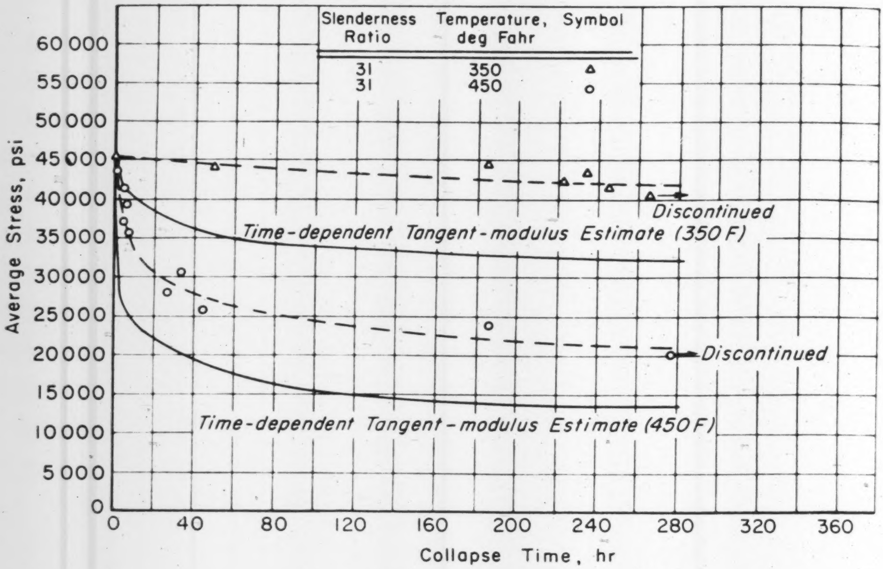

FIG. 4.—Average Stress-*versus*-Collapse Time for 2024-T4 Aluminum Alloy Columns.

beyond 4 hr. Although it is somewhat difficult to fix the time of the inflection points from the curves of Fig. 1, they appear to occur in the vicinity of approximately 5 hr. It appears then, that the accelerated creep rates beyond this may be associated with the loss in strength accompanying aging.

## SHORT-COLUMN TESTS

Tests were conducted on a column with a square $\frac{7}{16}$-in. cross-section. A slot was cut into the ends of each specimen, and hardened steel inserts were used to transmit the column load. V-notches were cut in the inserts and the distance between the root of the notches at each end of the specimen was the column length. By this measurement the column length used was 3.9 in.

The results of the column creep-buckling tests for the 3.9-in. column, whose computed slenderness ratio was 31, are presented in Fig. 4. The dashed curves were included to indicate the trend of the data. As might be anticipated, the decrease in capacity with time is much more drastic at 450 F than at 350 F. The marked decrease in the resistance to creep in this temperature range can also be observed in Figs. 1 and 2.

In a problem such as column-creep buckling, it is desirable to be able to predict the complex behavior encountered by the use of basic data such as uniform compression creep. In previous creep-buckling studies (**3**, **11**) the so-called time-dependent tangent-modulus method suggested by Shanley (**12**) has been used to obtain column-capacity estimates by the use of basic creep data.

The time-dependent tangent-modulus method is based on the use of isochronous stress-strain curves. Using the compressive-creep data of Figs. 1 and 2, isochronous stress-strain curves were constructed. The column average stress, $\sigma$, corresponding to the slenderness ratio of $\frac{L}{r} = 31$ were obtained by the use of the Euler formula,

$$\sigma = \frac{\pi^2 E_T(t)}{\left(\frac{L}{r}\right)^2},$$

where $E_T(t)$ is the time-dependent tangent-modulus. The results of these computations are presented as solid curves in Fig. 4.

It is apparent from a comparison of the capacity estimates and the column data that the method is markedly conservative for the test results.

Before passing final judgment on the method, however, it should be noted that since the hardened inserts were carefully centered, the column imperfections for the test specimens were probably quite small. A significant increase in imperfections could be expected to decrease the column capacities significantly.

For columns of this same alloy with $\frac{L}{r} = 81.2$, it has been observed (**13**) that an increase in imperfection[6] of from $\frac{L}{1000}$ to $\frac{L}{350}$ produced a decrease in capacity of the order of 10 per cent. Imperfections greater than $\frac{L}{350}$ would, in turn, be expected to result in additional decreases in capacities.

In view of the decrease in capacity that may be anticipated with the occurrence of imperfections larger than those present for the tests of Fig. 4, the tendency of the time-dependent tangent-modulus method to provide a conservative estimate may be interpreted as an advantage.

If imperfections become sufficiently large, the method can be expected to yield a nonconservative estimate. This has been shown to be possible in a previous paper (**11**). It has been recently shown (**13**), however, that for slender columns of a number of structural alloys, the estimate is conservative for imperfections less than about $\frac{L}{500}$. This is of interest because it suggests that the estimate can be expected to be fairly accurate in the range of imperfections normally encountered in structures.

Obviously, the time-dependent tangent-modulus method possesses limitations that are common to any approximate method. It does not, for example, provide for variations in imperfection, and it can be either conservative or nonconservative. It should be noted, however, that a method that can account for imperfection will undoubtedly be relatively complex. This added complexity will at least partially offset any advantage otherwise gained.

## CONCLUSIONS

Compression-creep curves for the aluminum alloy 2024-T4 exhibit decreasing creep rates during the initial stage of the tests and increasing creep rates during the final stage. A review of the aging characteristics of this alloy for the test temperatures indicates that the accelerated creep rate is probably associated with the loss in strength that accompanies aging.

Creep-buckling tests on short aluminum alloy 2024-T4 columns with a slenderness ratio of 31 indicate that column capacity decreases markedly in going from 350 F to 450 F. Column-capacity estimates based on Shanley's time-dependent tangent-modulus method are markedly conservative for the short-column data. It is noted, however, that columns with larger imperfections than those for the tested columns would have lower capacities, and that the accuracy could be expected to improve. Reference to other applications indicates that the estimate is good for a number of structural materials in the range of imperfections normally encountered.

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[6] Column imperfection (initial curvature, loading eccentricity) is frequently given in terms of column length, $L$. See reference (**14**).

*Acknowledgment:*

The work summarized in this paper was sponsored by the Materials Laboratory, Directorate of Research, Wright Air Development Center, under Contract No. AF 33(038)9542.

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## REFERENCES

(1) N. J. Hoff, "High-Temperature Effects in Aircraft Structures," *Applied Mechanics Reviews*, Vol. 8, No. 11, p. 453 (1955).

(2) E. E. Mathauser, and W. A. Brooks, "An Investigation of the Creep Lifetime of 75S-T6 Aluminum-Alloy Columns," *Technical Note* 3204, Nat. Advisory Committee for Aeronautics (1954).

(3) R. L. Carlson and G. K. Manning, "Investigation of Compressive-Creep Properties of Aluminum Columns at Elevated Temperatures," *Technical Report 52-251*, Wright Air Development Center, Part 2 (1954), Part 3 (1955), and Part 4 (1955).

(4) A. H. Sully, G. N. Cole, and G. Willoughby, "Creep of Metals Subjected to Compression Stress," *Nature*, Vol. 162, p. 411 (1948).

(5) J. N. Greenwood and H. K. Warner "Types of Creep Curve Obtained With Lead and Its Dilute Alloys," *Journal*, Inst. Metals, Vol. 64, p. 135 (1939).

(6) E. S. Machlin, "Creep Rupture by Vacancy Condensation," *Journal of Metals*, February, 1956. Vol. 8, No. 2, p. 106.

(7) C. Chen and E. S. Machlin, "Factors Affecting the Elevated Temperature Strength and Ductility in Metals," *Fifth Quarterly Progress Report*, Wright Air Development Center, Contract No. AF33(616)-2784 (1956).

(8) W. L. Finlay and W. R. Hibbard, "Some Effects of Applied Stresses on Precipitation Phenomena," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., p. 255 (1949).

(9) E. H. Dix, "New Developments in High-Strength Aluminum Alloy Products," *Transactions*, Am. Soc. for Metals, Vol. 35, p. 130 (1945).

(10) E. E. Underwood, "Creep of Aluminum-Copper Alloys During Age Hardening," to be published in *Transactions*, Am. Inst. Mining, Metallurgical, Engrs.

(11) R. L. Carlson, "Time-Dependent Tangent Modulus Applied to Column-Creep Buckling," *Transactions*, Am. Soc. Mechanical Engineers, *Journal of Applied Mechanics*, Vol. 23, No. 3 (1956).

(12) F. R. Shanley, "Weight-Strength Analysis of Aircraft Structures," McGraw-Hill Book Co., Inc., New York, N. Y., Chapt. 19 (1952).

(13) R. L. Carlson and G. K. Manning, "A Summary of Compressive-Creep Characteristics of Metal Columns at Elevated Temperatures," *Technical Report 57-96*, Wright Air Development Center, March, 1957.

(14) S. Timoshenko, "Theory of Elastic Stability," McGraw-Hill Book Co., Inc., New York, N. Y., p. 186 (1936).

# DISCUSSION

MR. JOSEPH MARIN.[1]—Did the authors express the tension creep data and the compression creep data by two different equations and then use these relations in determining an equation for columns? In their interpretation of creep in columns did they consider all the various available theories for this purpose?

MR. R. L. CARLSON (*author*).—All of the creep data that we presented here is for compressive loading only. Although unloading on the concave face could be expected, the stresses would be compressive throughout for the values of slenderness ratio studied. Tensile data would not, therefore, be required.

We have examined methods that have been suggested in the literature in terms of their applicability. Unfortunately, most of these methods had limitations that restrict their use. For example, the use of a viscoelastic element to represent creep response does not provide a good enough representation of true behavior in the temperature range studied to be applicable. More complex empirical relations that do provide a more satisfactory representation of the creep response are readily applicable only to model columns, that is, columns with cross-sections consisting of two flanges and a web of zero thickness. Solutions for columns with a solid cross-section are quite lengthy and they are accomplished by tedious step-by-step type computations. When these difficulties are added to the fact that an understanding of creep response to stresses that vary with time has not reached a definitive stage, we do not feel that the labor involved is justified. In essence, we feel that a problem such as column creep buckling is not a good one for "testing" the validity of creep laws. Simpler experiments that would provide more decisive judgments can be devised.

The method of analysis used in the paper provides only approximate values of column capacity. It is not considered by any means a "final" solution; it is to be expected that improved methods will be evolved. Until then, however, it makes available a simple procedure that is based on actual creep behavior, and its relationship to true column capacity is understood (see reference (11) of paper).

MR. MAXWELL PEVAR.[2]—Can you briefly describe this time dependent method for determining modulus?

MR. CARLSON.—A detailed discussion of the use of this method and how it is related to the actual column capacity is presented in reference (11) of the paper. Since this reference is readily available, I would prefer not to repeat the details here.

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[1] Professor and Head, Department of Engineering Mechanics, College of Engineering and Architecture, Pennsylvania State University, University Park, Pa.

[2] Engineering Physicist, Airframe Research Department, The Budd Co., Philadelphia, Pa.

# TENSILE AND CREEP PROPERTIES OF TUNGSTEN AT ELEVATED TEMPERATURES*

By J. W. Pugh[1]

Of all metals, tungsten has the highest melting point (6170 F). It might be expected to have useful mechanical strength at very high temperatures and has, in fact, demonstrated this strength in a few specialized applications. Despite its long application in the lamp industry, the tensile characteristics of tungsten have not been well documented. Tensile strength of wire (**1**)[2] and tensile strength of single-crystal wire (**2**) as a function of temperature were determined many years ago. Strengths obtained in these cases were exceedingly high; cold-drawn wire at room temperature had a tensile strength of about 500,000 psi. More recently, the tensile properties of annealed bar stock have been examined at temperatures up to 1650 F (**3**). The present investigation adds considerably to this information in the range from room temperature to 2200 F.

## Preparation of Material

A sintered tungsten ingot having a 1-in. square cross-section was used. This ingot was made at the Cleveland Wire Plant of the General Electric Co. by a standard production technique. It was swaged in the Research Laboratory according to the following schedule:

| Temperature, deg Fahr. | Initial Size | Final Size |
|---|---|---|
| 2730 | 1 in. square | 0.900 in. round |
| 2550 | 0.900 in. round | 0.700 in. round |
| 2370 | 0.700 in. round | 0.600 in. round |
| 2190 | 0.600 in. round | 0.500 in. round |
| 2010 | 0.500 in. round | 0.345 in. round |

Part of the swaged rods were annealed at 2900 F and the remainder were annealed at 1830 F. Both treatments were for 1 hr. The first treatment produced a recrystallized structure having an average grain diameter of 0.045 mm (see Fig. 1). The 1830 F treatment resulted only in stress relief. Figure 2, a photomicrograph of this structure, is identical in appearance to photomicrographs of as-swaged rod. The furnace atmosphere for sintering, swaging and annealing was, in each case, hydrogen.

Chemical analysis obtained from a specimen of the swaged rod gave the following results:

| Element | Percentage by Weight |
|---|---|
| Hydrogen | $0.0000 + 0.0001, - 0.0000$[a] |
| Carbon | $0.01 \pm 0.01$[a] |
| Nitrogen | $0.003 \pm 0.002$[b] |
| Oxygen | $0.0131 \pm 0.0009$[a] |
| Silicon | nil |
| Aluminum | trace |
| Calcium | trace |
| Sodium | nil |
| Iron | trace |
| Nickel | trace |
| Potassium | trace |
| Copper | nil |
| Thorium | nil |

[a] Vacuum Fusion Analysis.
[b] Kjeldahl Analysis.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] General Electric Lamp Division, Cleveland, Ohio; formerly with General Electric Research Laboratory, Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 915.

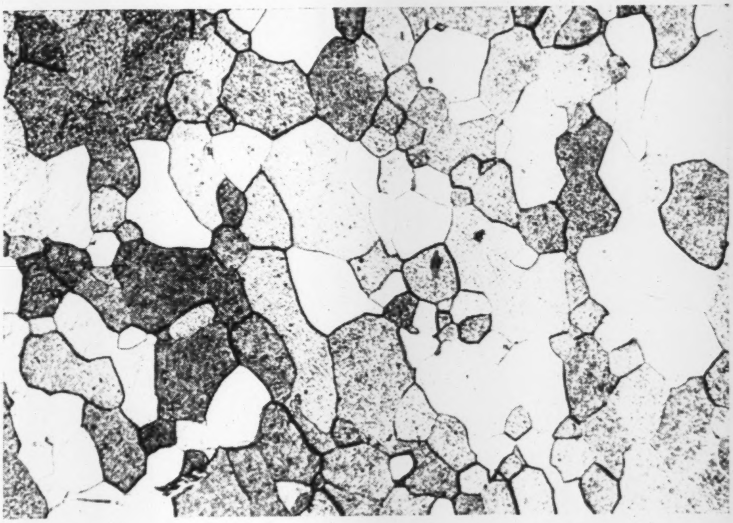

FIG. 1.—Photomicrograph of Swaged Tungsten Rod Annealed 3 hr at 2900 F (×250). Etched in NaOH + $K_3Fe(CN)_6$.

FIG. 2.—Photomicrograph of Swaged Tungsten Rod Annealed 3 hr at 1830 F (×500). Etched in NaOH + $K_3Fe(CN)_6$.

## TESTING PROCEDURE

Short-time tension tests were made using an Instron testing machine at the rates 0.2 and 0.02 in. per in. per min. Flow curves were recorded autographically. Specimens were protected from atmospheric contamination by testing them in a vacuum capsule. Temperatures were measured by means of a platinum - platinum rhodium thermocouple resting on top of the specimen. The specimen used is illustrated in Fig. 3.

Constant load creep rupture tests were conducted in an argon atmosphere at 1600, 1800, 2000, and 2200 F. The machine used employed a test string which, together with strain measuring devices, is enclosed in a vacuum tight capsule. Otherwise it is an ordinary constant load machine with a 20:1 lever ratio. Specimens used were identical to those used for the short-time tension test (Fig. 3) except that threads were ground on the ends for attaching strain measuring rods.

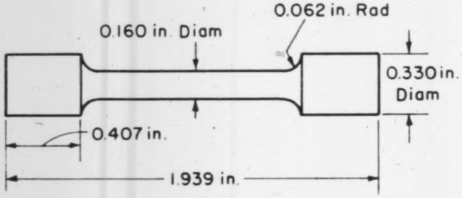

FIG. 3.—Tension Specimen.

More detailed descriptions of these testing devices can be found elsewhere (**4**).

## RESULTS AND DISCUSSION

### *Short-Time Tension Tests:*

True stress-strain curves for recrystallized tungsten are shown in Fig. 4. These curves for temperatures below 1600 F can be fairly accurately described by the equation

$$\sigma = A\epsilon^m \Big|_{\dot{\epsilon}, T}, \ldots\ldots\ldots\ldots (1)$$

where:

$\sigma$ = stress,
$A$ = stress at unit strain,
$\epsilon$ = strain,
$m$ = strain-hardening exponent,
$T$ = temperature, and
$\dot{\epsilon}$ = strain rate.

Above 1600 F there is a deviation from this expression at strains below about 0.1. The strain-hardening exponent $m$ was evaluated at the necking strain where Eq. 1 is valid in all cases. Rate sensitivity was evaluated by comparing the flow stress at two different rates and constant strain. It is defined by

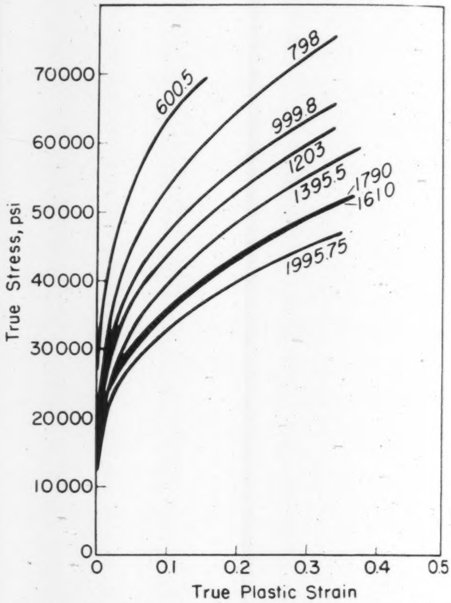

FIG. 4.—Flow Curves for Recrystallized Tungsten.

$$n = \frac{\log (\sigma_1/\sigma_2)}{\log (\dot{\epsilon}_1/\dot{\epsilon}_2)}\Bigg|_{\epsilon, T}, \ldots\ldots\ldots (2)$$

where:

$n$ = strain-rate sensitivity exponent,
$\sigma_1$ = stress at the fast rate,
$\sigma_2$ = stress at the slow rate,
$\dot{\epsilon}_1$ = the fast strain rate = 0.2 per min, and
$\dot{\epsilon}_2$ = the slow strain rate = 0.02 per min.

Little change of either rate sensitivity or strain hardening is observed for the temperature range covered. The strain hardening exponent at 2000 F is larger than that at 600 F. Actually, if the stress-strain curves are compared (Fig. 4) the strain hardening is obviously less at 2000 F. An evaluation of the slopes of the curves in Fig. 4 at $\epsilon = 0.1$ gives a

TABLE I.—TENSION TEST PARAMETERS.

| Treatment | Temperature, deg Fahr | Yield Strength, 0.02 offset, per cent | Ultimate Tensile Strength, psi | Elongation, per cent | Strain Hardening Coefficient, $m$ | Strain Rate Sensitivity Exponent, $n$ | Slope of $\sigma - \epsilon$ curve at $\epsilon = 0.1$ psi |
|---|---|---|---|---|---|---|---|
| Annealed 1 hr at 2900 F... | 202 | ... | 79 600 | ... | ... | ... | |
| | 600 | 28 800 | 59 700 | 16.4 | 0.244 | ... | 153 000 |
| | 798 | 22 000 | 54 100 | 62.4 | 0.299 | 0.0104 | 160 000 |
| | 1000 | 17 900 | 47 200 | 51.0 | 0.288 | 0.0061 | 164 000 |
| | 1203 | 15 200 | 44 500 | 55.3 | 0.292 | 0.0065 | 127 000 |
| | 1395 | 13 000 | 41 200 | 57.1 | 0.308 | 0.0053 | 129 000 |
| | 1610 | 14 500 | 36 600 | 57.9 | 0.327 | 0.0072 | 116 000 |
| | 1790 | 14 400 | 36 500 | 56.8 | 0.304 | 0.0066 | 111 000 |
| | 1995 | 12 900 | 33 500 | 52.0 | 0.306 | 0.0038 | 81 000 |
| Annealed 1 hr at 1830 F... | 80 | ... | 215 000 | 0.2 | ... | ... | ... |
| | 206 | ... | 145 000 | 3.3 | ... | ... | ... |
| | 309 | ... | 146 000 | 26.4 | ... | ... | ... |
| | 405 | ... | 125 400 | 30.0 | ... | ... | ... |
| | 599 | ... | 103 000 | 24.5 | ... | ... | ... |
| | 798 | ... | 85 100 | 24.5 | ... | ... | ... |
| | 1007 | ... | 80 800 | 18.2 | ... | ... | ... |
| | 1195 | ... | 76 579 | 16.0 | ... | ... | ... |
| | 1408 | ... | 77 900 | 13.6 | ... | ... | ... |
| | 1600 | ... | 66 200 | 13.5 | ... | ... | ... |
| | 1803 | ... | 61 500 | 15.5 | ... | ... | ... |
| | 2000 | ... | 58 969 | 14.5 | ... | ... | ... |

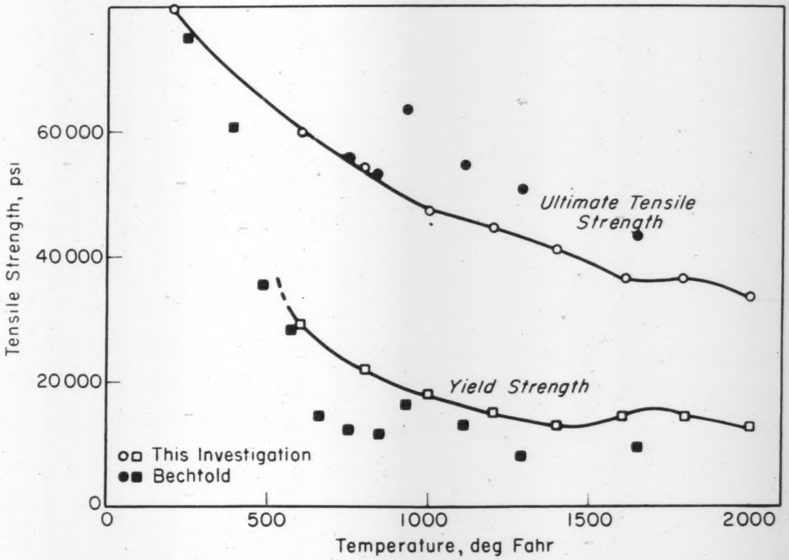

FIG. 5.—Ultimate Tensile Strength and Yield Strength as a Function of Temperature for Recrystallized Tungsten.

considerably different comparison than that of the more common parameter $m$ (see Table I). This illustrates the inconsistency of relying on the value of $m$ when comparing curves having radically different flow stresses.

Figure 5 shows the temperature dependence of tensile strength and yield

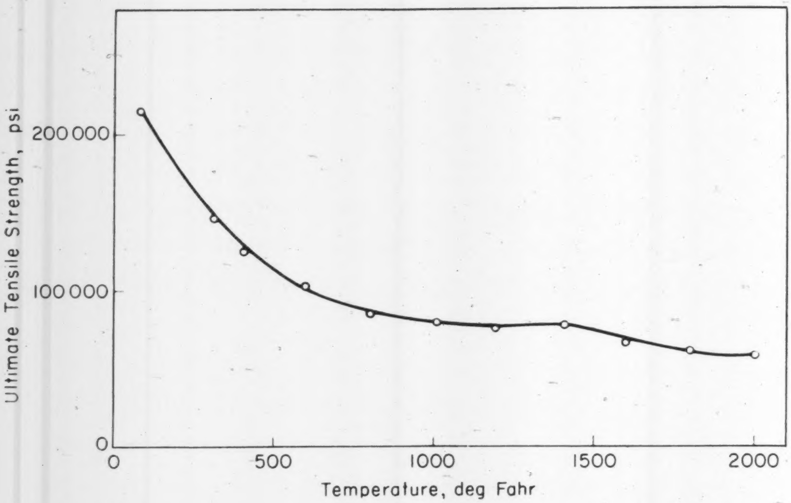

FIG. 6.—Ultimate Tensile Strength as a Function of Temperature for Unrecrystallized Tungsten

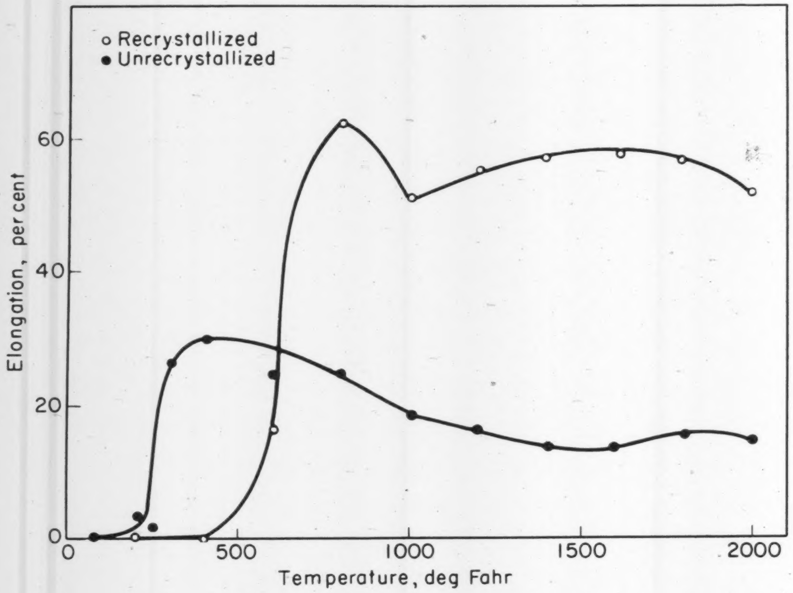

FIG. 7.—Elongation as a Function of Temperature.

strength for tungsten as recrystallized. Bechtold and Shewmon's data (**3**) are plotted on the same coordinates for comparison. Their data were obtained at the somewhat slower strain rate of 0.0148 in. per in. per min. Bechtold and Shewmon attribute the 930 F peaks in their data to precipitation hardening. It is also possible that they are due to strain aging.

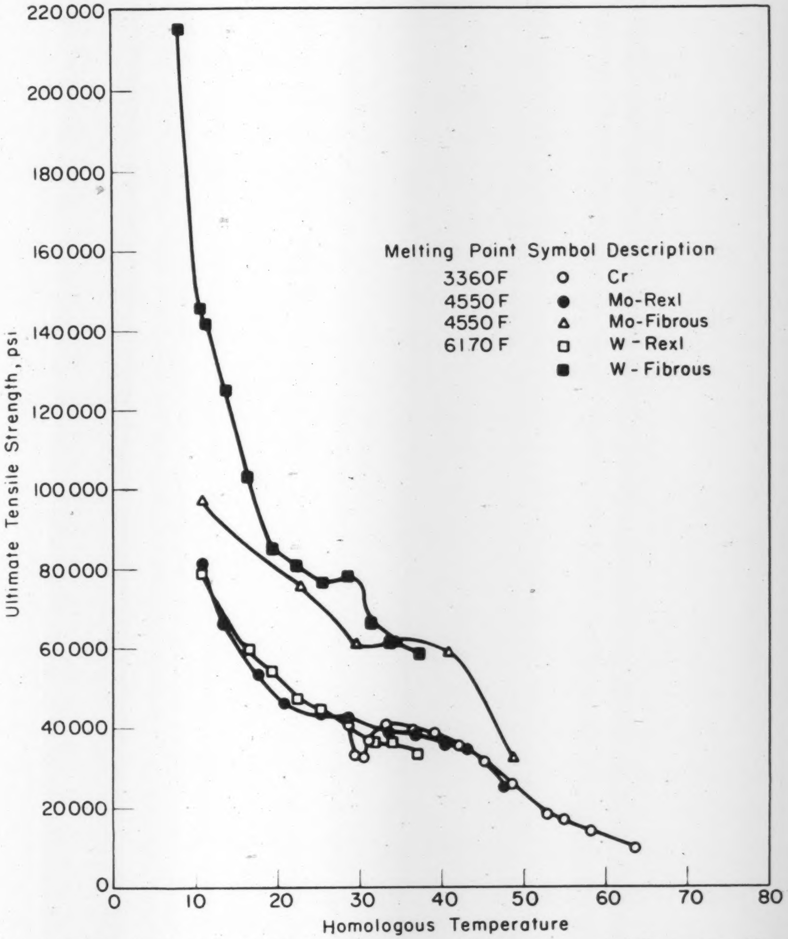

FIG. 8.—A Comparison of Strength for Chromium, Molybdenum, and Tungsten. Homologous Temperature is Testing Temperature in per cent of the Melting Point.

Figure 6 shows the temperature dependence of strength for unrecrystallized tungsten as stress-relief-annealed. Yield strength data are not included because they are not significantly different than the ultimate or maximum strength. The strength represented here is 50 to 100 per cent higher than the strength of the recrystallized metal.

Per cent elongation data for both conditions are presented in Fig. 7. The ductile-brittle transition temperature appears to be 600 F for the recrystallized tungsten and 300 F for the stress-relief-annealed tungsten.

It is not clear to what extent the rela-

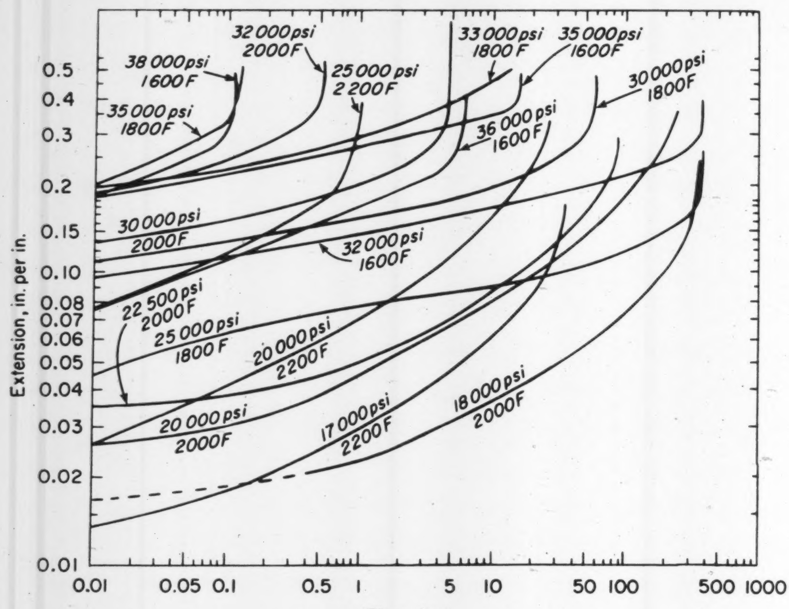

FIG. 9.—Logarithmic Strain-Time Curves for Recrystallized Tungsten.

tively good strength of tungsten is related to its very high melting point. In Fig. 8 the temperature dependence of the strength of tungsten is compared with that of two other body-centered cubic refractory metals which melt at very different temperatures. The molybdenum data were obtained for recrystallized molybdenum of the same grain size as the tungsten (5). Chromium data are for a hot-worked structure having a grain size of approximately 0.5 mm (6). Test conditions were identical in all three cases. Comparison is made on a homologous temperature scale which describes the test temperature as a percentage of the melting point of the metal. Observe that the contribution to strength caused by the fibrous (cold-worked) structure is considerable for both tungsten and molybdenum. The contribution to the strength of tungsten is, however, more significant at the lower temperatures. This comparison is obscured at higher temperatures by a strain-aging effect in both metals. In the recrystallized condi-

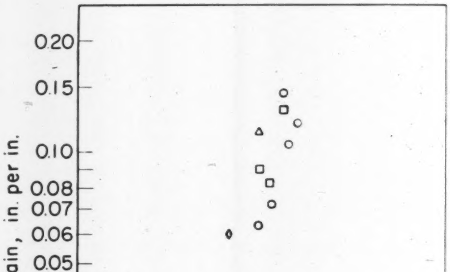

FIG. 10.—Loading Strain as a Function of Stress for Recrystallized Tungsten.

tion the three metals compare quite favorably on the homologous temperature scale.

All the short-time tensile properties evaluated are summarized in Table I.

*Creep-Rupture Tests:*

The creep curves obtained for recrystallized tungsten are presented in Fig. 9 in logarithmic form, so that they might all be included on a single set of coordinates. The initial strain encountered on loading each specimen is presented as a function of stress in Fig. 10. It indicates that the stress range is of considerably more importance to initial strain than the temperature range for these tests. Figure 11 shows the correlation of initial stress *versus* rupture life for these tests.

Table II summarizes the rupture life properties for tungsten and provides a comparison with two other materials, molybdenum (5) and Jetalloy 1570 (7). Jetalloy 1570 is a new cobalt-nickel-chromium base alloy which has outstanding mechanical properties and is generally superior to current commercial alloys for high-temperature, high-stress applications such as turbine buckets. Relative slopes of the stress-rupture life curves of the three materials are listed in Table III. Note that the initial slopes of these plots were smaller for tungsten in each case. It was not determined whether the fracture mode changed from transcrystalline to intercrystalline at the points where the logarithmic stress-life plots changed slope.

Minimum creep rates are plotted as a function of stress in Fig. 12. This parameter appears to be somewhat more responsive to changes in stress at 1600 F than at 2200 F. Put another way, the

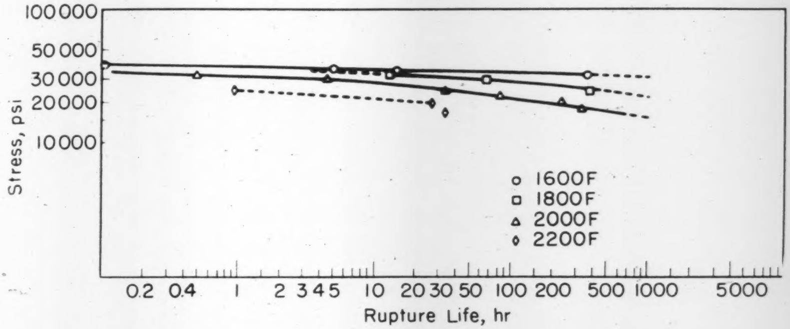

FIG. 11.—Stress-Rupture Properties for Recrystallized Tungsten.

TABLE II.—STRESS RUPTURE PROPERTIES.

| Temperature, deg Fahr | Metal | 10 hr, psi | 100 hr, psi | 1000 hr, psi |
|---|---|---|---|---|
| 1600 | Tungsten | 35 000 | 33 000 | 31 000 |
| | Molybdenum | 28 000 | 22 000 | 17 000 |
| | Jetalloy 1570 | 32 500 | 22 500 | 15 500 |
| 1800 | Tungsten | 33 000 | 28 500 | 22 000 |
| | Molybdenum | 19 000 | 15 000 | 12 000 |
| 2000 | Tungsten | 29 500 | 22 000 | 15 500 |
| | Molybdenum | 14 500 | 11 500 | 9 000 |
| 2200 | Tungsten | 21 500 | | |

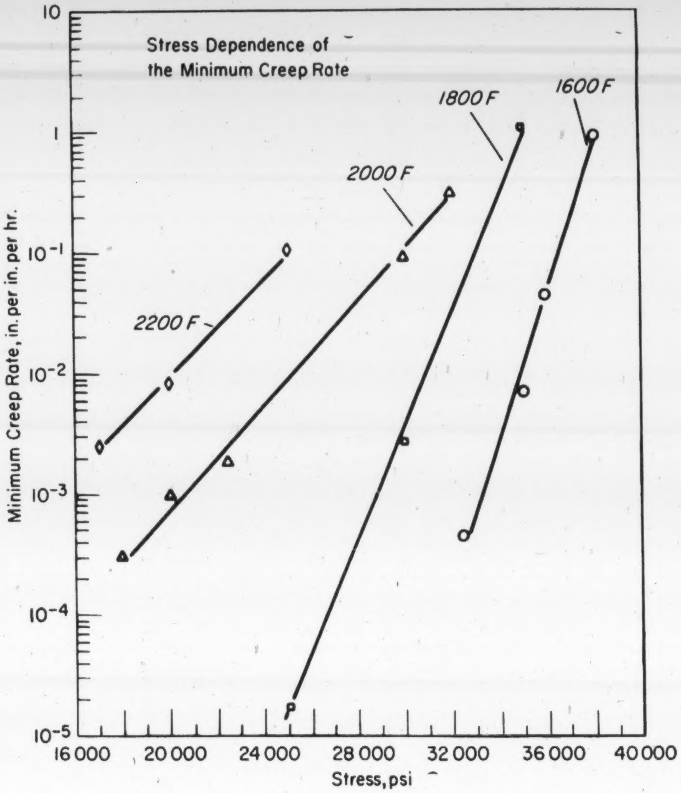

Fig. 12.—Minimum Creep Rate as a Function of Stress for Recrystallized Tungsten.

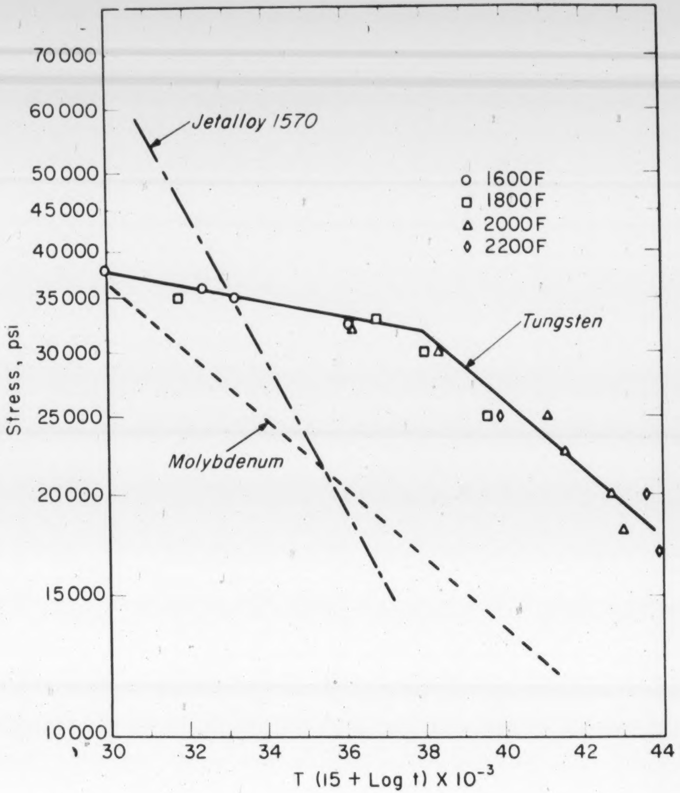

Fig. 13.—Stress as a Function of the Larson-Miller Parameter for Recrystallized Tungsten.

strain rate sensitivity (that is the dependence of flow stress on strain rate) is higher at the higher temperatures. Note that the 1600 and 1800 F curves are approximately parallel and that the 2000 and 2200 F curves seem also to be parallel but have different slope than the 1600 and 1800 F curves. This suggests a change in the character of the recovery process between 1800 and 2000 F. In this connection, X-ray diffraction powder patterns have been made for cold-rolled commercial grade tungsten after a series of annealing treatments. It was observed that the Debye rings first became spotty for temperatures in the range between 1800 and 2000 F (8). This suggests that polygonization or incipient recrystallization takes place at this temperature and may account for the difference in slopes observed in Fig. 12.

TABLE III.—SLOPES OF LOGARITHMIC STRESS-RUPTURE LIFE CURVES.

| Temperature, deg Fahr | Tungsten | Molybdenum | Jetalloy 1570 |
|---|---|---|---|
| 1600 ...... | 0.0141 | 0.106 | 0.166 |
| 1800 ...... | 0.0165 to 0.109 | 0.100 | ... |
| 2000 ...... | 0.0291 to 0.160 | 0.104 | ... |
| 2200 ...... | 0.0638 | ... | ... |

Figure 13 is a plot of stress *versus* the Larson-Miller parameter (9). Molybdenum (5) and Jetalloy 1570 (7) are again included for comparison. Molybdenum is obviously inferior above a value of 30,000 for the parameter while Jetalloy 1570 is inferior above 33,000.

## SUMMARY

Tungsten has been shown to be stronger than other representative refractory metals in short-time tension tests from room temperature to 2000 F. The margin of its superiority in this respect is proportional to its higher melting point. Tungsten's creep-rupture properties are more remarkable by comparison with commercial austenitic alloys and molybdenum in the range 1600 to 2200 F. Many high-temperature applications depend on strength-to-weight ratio and in this respect tungsten is less outstanding. However, tungsten is slightly superior to molybdenum even in strength-weight ratio at 2000 F. Furthermore, it is highly probable that tungsten's advantage above 2000 F would increase rapidly with temperature. These considerations indicate that tungsten's future as an ultra-high-temperature metal is somewhat brighter than anticipated.

*Acknowledgment:*

Credit is due to W. E. Rollins for taking the experimental data and to Miss B. J. Drummond for reducing the data to tensile parameters.

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## REFERENCES

(1) F. S. Goucher, "On the Strength of Tungsten Single Crystals and its Variation with Temperature," *Philosophical Magazine*, Vol. 48, p. 229 (1924).

(2) F. S. Goucher, "Further Studies on the Deformation of Tungsten Single Crystal," *Philosophical Mag.*, Vol. 2, p. 289 (1926).

(3) J. H. Bechtold and P. G. Shewmon, "Flow and Fracture Characteristics of Annealed Tungsten," *Transactions*, Am. Soc. Metals, Vol. 46, p. 397 (1954).

(4) J. W. Pugh, "How to Test Refractory Metals," *Steel*, Vol. 137, No. 16, Oct. 17, 1955, p. 114.

(5) J. W. Pugh, "The Tensile Properties of Molybdenum at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 47, p. 984 (1955).

(6) J. W. Pugh, General Electric Research Lab., RL-1040, Dec. 1953.

(7) R. W. Guard and T. A. Prater, "An Austenitic Alloy for High Temperature Use," To be published.

(8) J. W. Pugh, Unpublished Data (1956).

(9) F. R. Larson and J. Miller, "A Time Temperature Relationship for Rupture and Creep Stresses," Am. Soc. Mechanical Engrs., *Paper No. 51-A-36*, Annual Meeting, Nov. 1951.

# DISCUSSION

Mr. Richard S. Cremisio.[1]—I would like to inquire as to the material used for pull rods in this investigation.

Mr. J. W. Pugh (*author*).—A variety of materials have been used in this equipment ranging from S-590 and Inconel to molybdenum and molybdenum alloys. To the best of my knowledge, an alloy of molybdenum plus 0.3 per cent columbium was used for the tests which are described here.

A description of the test equipment has appeared recently in *Steel* (reference (4) of the paper).

Mr. Frank Burkett.[2]—Do you have any fatigue data either at room temperature or high temperature for tungsten?

Mr. Pugh.—I have not yet made any fatigue tests.

Chairman H. C. Cross.[3]—What is contemplated for the protection of tungsten for possible high-temperature applications? We are familiar with the problem as regards molybdenum and molybdenum-base materials, but what about tungsten-base materials?

Mr. Pugh.—I have not yet attacked this problem, nor has anyone in our laboratory. We know of course that tungsten is more oxidation resistant than molybdenum. For example it does not oxidize catastrophically below at least 2200 F. We also know that molybdenum disilicide coating techniques may be applied to tungsten. The tungsten disilicide coating can be readily produced by catalytic reduction of silicon tetrachloride and is said to give prolonged protection up to 1500 C. This is approximately 100 C higher than molybdenum disilicide is effective. However such coatings appear to be even more brittle than the molybdenum disilicide coatings at room temperature.[4]

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[1] Staff Metallurgist, Crucible Steel Company of America, Research Laboratories, Pittsburgh, Pa.

[2] Metallurgical Laboratory, The Dow Chemical Co., Midland, Mich.

[3] Metallurgist and Assistant Coordination Director, Battelle Memorial Inst., Columbus, Ohio.

[4] E. Fitzer, "Materials of Maximum Creep and Oxidation Resistance Produced by Coating Tungsten and Molybdenum with Vapor Deposited Silicon," *Berg und Hüttenmannische Monatshefte*, Vol. 97, No. 5, p. 81 (1952).

# DYNAMIC ELASTIC MODULUS VALUES AT HIGH TEMPERATURES FOR NICKEL-BASE, ALUMINUM-BASE, AND METAL-METAL OXIDE ALLOYS*

By Robert F. Wilde[1] and Nicholas J. Grant[2]

## Synopsis

The elastic modulus was measured by a dynamic method for heat-resistant nickel-base alloys from 80 to 1600 F; for type 316 stainless steel from 80 to 1600 F; for pure aluminum and a number of high-purity solid solution alloys from 80 to 800 F; and for a number of sintered, extruded metal-metal oxide alloys based on copper and on nickel.

The purpose of this investigation was to determine the elastic modulus at elevated temperatures for a number of materials currently being studied in this laboratory (see Table I). A dynamic method known as the "free-free" or floating beam method was used; it has been amply described by others (**1**, **2**, **3**).[3] Since the requirements and theory for the test are known, they will not be repeated. Briefly, a uniform rod is vibrated in transverse flexure and the resonant frequency is determined. This frequency is dependent on the elastic modulus and the physical constants of the specimen. The mathematical relationship which is obeyed is $E = Kf^2$, where $K$ includes the physical constants of the specimen, and $f$ is the fundamental resonant frequency. Because the dimensions and mass of the specimen are easily determined with sufficient accuracy and electronic frequency measurements are precise, the main problem faced in this method was whether the frequency being measured was the true resonant frequency of the bar. Several sources of error must be considered. The specimen and its supporting and driving transducers can be considered as an undamped, coupled spring system. It can be shown that a system of this type possesses two resonant frequencies. These frequencies and the amplitude of vibration accompanying them can be varied and controlled by proper choice of the coupling and the relative masses of the spring systems. By keeping the coupling small (but not negligible, otherwise the driven system would not vibrate) and the mass of the driving system small compared to that of the specimen or driven system, the frequencies will be well separated and the amplitude of the unwanted frequency will be small. Thus, the desired frequency, that of the specimen vibrating freely, can be approached quite closely. The mode of vibration of the specimen can be solved by using a feeler to determine whether the specimen is vibrating at the fundamental frequency.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Formerly Research Assistant, Department of Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass., now, General Electric Co., Lynn, Mass.

[2] Professor, Department of Metallurgy, Massachusetts Institute of Technology, Cambridge, Massachusetts.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 925.

TABLE I.—COMPOSITIONS, PER CENT, AND TEMPERATURE RANGE OF TESTS.

NICKEL-BASE HEAT-RESISTANT ALLOYS (80 TO 1600 F)

| | Cr | Co | Mo | Ti | Al | Cb | C | Ni |
|---|---|---|---|---|---|---|---|---|
| Inconel X-550[a] | 15.2 | ... | ... | 2.50 | 1.17 | 0.97 | 0.04 | balance |
| Inco 700[a] | 15.1 | 30.7 | 3.2 | 2.19 | 3.02 | ... | 0.13 | |
| Waspalloy[b] | 19.7 | 14.1 | 4.2 | 3.25 | 1.39 | ... | 0.09 | |
| M-252[b] | 19.7 | 9.6 | 9.9 | 2.52 | 1.04 | ... | 0.12 | |
| 6 V[c] | 18.9 | ... | ... | 5.88 | ... | ... | 0.006 | |
| 8 V[c] | 20.7 | ... | ... | 2.98 | 1.43 | ... | 0.04 | |
| 11 V[c] | 19.8 | ... | ... | 3.85 | 1.38 | ... | 0.03 | |

AUSTENITIC STAINLESS STEEL (80 TO 1600 F)

Type 316

HIGH-PURITY ALUMINUM AND ALLOYS (80 TO 800 F)

Al-5 per cent Zn (2 phase below about 250 F)
Al-20 per cent Zn (2 phase below about 465 F)
Al-0.94 per cent Mg (single phase down to 80 F)
Al-5.1 per cent Mg (2 phase below about 480 F)
Al-0.24 per cent Cu (2 phase below about 480 F)
Al-2.0 per cent Cu (2 phase below about 700 F)

METAL-METAL OXIDE ALLOYS (SINTERED AND EXTRUDED) (80 TO 800 F)

−200 mesh copper with 0, 1, and 3 per cent $Cu_2O$ (by volume)
−200 mesh copper with 1, 3, and 10 per cent $SiO_2$ ($<10\ \mu$) (by volume)
−200 mesh copper with 1, 3, and 10 per cent $Al_2O_3$ ($<10\ \mu$) (by volume)
−10 $\mu$ nickel with 12 and 25 per cent $Al_2O_3$ ($0.3\ \mu$) (by volume)

[a] Air melted
[b] Vacuum melted
[c] Vacuum melted, experimental heats

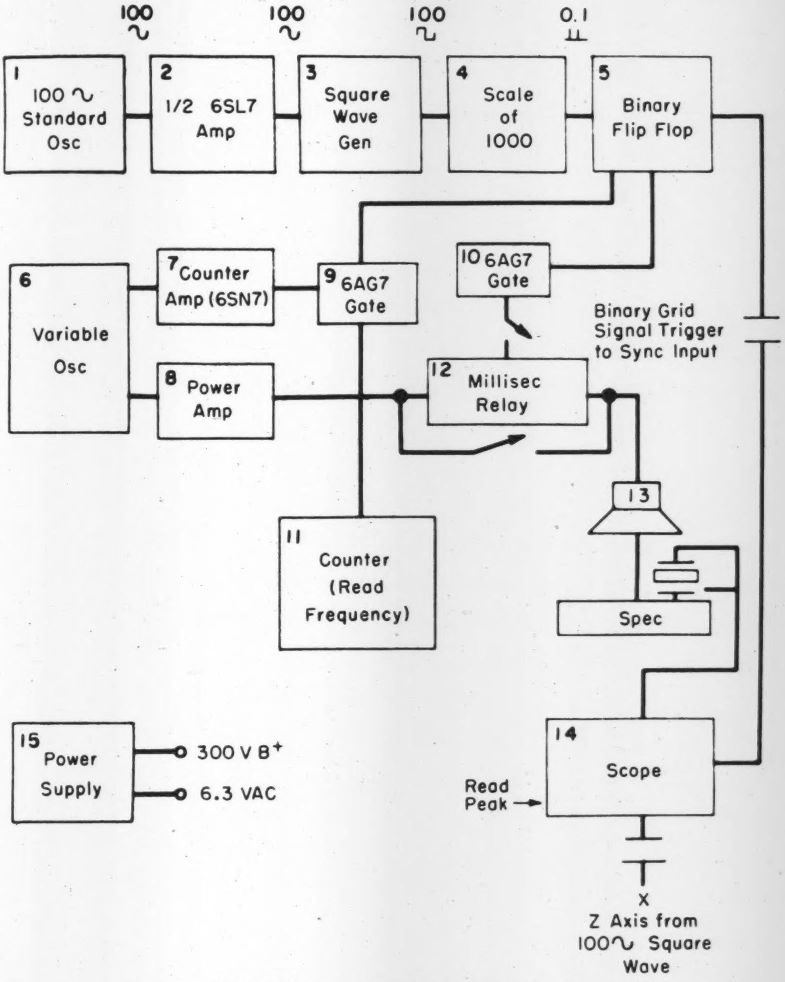
The change in specimen dimensions on heating is another variable. This change is significant only as regards the change in length since the change in diameter appears only as a ratio. Previous investigators have ignored the correction for thermal expansion, and it has been ignored in this work. When all of the sources of error in the dynamic measurement of the elastic modulus are evaluated, as Andrews has done (3), it can be seen that the thermal expansion correction is about 1 per cent at 1500 F, and the cumulative error from other sources is at least ±1 per cent. The change in modulus through correction for dimensions is shown only for alloy M-252 in Table III.

## MATERIALS INVESTIGATED

Alloys on which measurements were made and the test temperatures range are listed in Table I.

## EXPERIMENTAL METHOD

A simplified block diagram of the apparatus is shown in Fig. 1. The essential equipment is a variable frequency oscillator, a driving transducer, a receiving transducer, a frequency measuring device, and a furnace of suitable design. An oscilloscope is useful to insure that the waveform of the received signal is a pure sine wave and to follow the resonance peak as it changes frequency.


100 ∿
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0.1
1 100 ∿ Standard Osc
2 1/2 6SL7 Amp
3 Square Wave Gen
4 Scale of 1000
5 Binary Flip Flop
6 Variable Osc
7 Counter Amp (6SN7)
8 Power Amp
9 6AG7 Gate
10 6AG7 Gate
Binary Grid Signal Trigger to Sync Input
12 Millisec Relay
13
11 Counter (Read Frequency)
Spec
14 Scope
Read Peak
15 Power Supply
300 V B+
6.3 VAC
X
Z Axis from 100∿ Square Wave

Fig. 1.—Block Diagram of Elastic Modulus Apparatus.

The working formula from which the unrelaxed modulus was calculated is:

$$E\ (\text{psi} \times 10^7) = 2.0239 \times 10^{-8} \left(\frac{L}{d}\right)^4 \frac{(M)}{(L)} f^2$$

where:

$L$ = length in inches,
$d$ = diameter in inches,
$M$ = mass in pounds, and
$f$ = resonant frequency.

Cylindrical rods of each material were carefully machined and measured to within 0.001 in. and weighed to within $2.5 \times 10^{-6}$ lb (0.001 g). The resonant frequency could be determined to ±1 cps. Temperatures were accurately measured with a thermocouple.

Measurements at elevated temperatures were conducted by supporting the specimen by fine Chromel wires in a

### TABLE II.—ELASTIC MODULUS DATA.

| Material | Temperature, deg Fahr | Modulus of Elasticity, $E$, psi |
|---|---|---|
| **Nickel-Base Alloys** | | |
| Inconel X 550 | 80 | $29.8 \times 10^6$ |
| | 500 | 28.1 |
| | 1000 | 25.8 |
| | 1500 | 23.1 |
| | 1600 | 22.1 |
| Inco 700 | 80 | 31.9 |
| | 500 | 30.6 |
| | 1000 | 28.0 |
| | 1500 | 24.5 |
| | 1600 | 23.6 |
| Waspalloy | 76 | 30.6 |
| | 500 | 29.1 |
| | 1000 | 26.7 |
| | 1300 | 24.9 |
| | 1500 | 23.6 |
| | 1600 | 22.7 |
| 6V (6 per cent Ti) | 85 | 30.4 |
| | 400 | 29.1 |
| | 800 | 27.6 |
| | 1200 | 25.1 |
| | 1500 | 23.2 |
| | 1600 | 22.5 |
| 8V (3 per cent Ti, 1.4 per cent Al) | 85 | 30.8 |
| | 418 | 29.5 |
| | 800 | 27.9 |
| | 1200 | 25.8 |
| | 1500 | 23.9 |
| 11 V (4 per cent Ti, 1.4 per cent Al) | 80 | 30.7 |
| | 410 | 29.7 |
| | 800 | 28.2 |
| | 1200 | 26.1 |
| | 1500 | 24.2 |
| **Type 316 Stainless Steel** | | |
| 316 Stainless | 80 | 27.6 |
| | 500 | 25.3 |
| | 1000 | 22.6 |
| | 1500 | 19.5 |
| | 1600 | 19.0 |
| **High-Purity Aluminum-Base Alloys** | | |
| 5.0 per cent Zn | 85 | 9.77 |
| | 200 | 9.47 |
| | 400 | 8.91 |
| | 600 | 8.25 |
| | 800 | 7.66 |
| **High-Purity Aluminum-Base Alloys—*Continued*** | | |
| 20.0 per cent Zn | 85 | 9.38 |
| | 200 | 9.14 |
| | 400 | 8.59 |
| | 600 | 7.84 |
| | 800 | 7.12 |
| 0.94 per cent Mg | 85 | 9.69 |
| | 200 | 9.46 |
| | 290 | 9.15 |
| | 400 | 8.93 |
| | 600 | 8.28 |
| | 800 | 7.84 |
| 5.10 per cent Mg | 85 | 9.46 |
| | 145 | 9.26 |
| | 200 | 9.15 |
| | 402 | 8.60 |
| | 600 | 7.91 |
| | 800 | 7.17 |
| 0.2 per cent Cu | 85 | 9.72 |
| | 200 | 9.40 |
| | 400 | 8.92 |
| | 600 | 8.41 |
| | 800 | 7.89 |
| 2.0 per cent Cu | 85 | 9.78 |
| | 210 | 9.39 |
| | 405 | 8.85 |
| | 600 | 8.30 |
| | 800 | 7.78 |
| **Metal - Metal Oxide Alloys** | | |
| Cu-1 per cent $Al_2O_3$ | 85 | $17.8 \times 10^6$ |
| | 250 | 17.4 |
| | 400 | 17.2 |
| | 600 | 16.7 |
| (Cooling only) | 610 | 16.6 |
| | 800 | 16.1 |
| Cu-3 per cent $Al_2O_3$ | 80 | 10.9 |
| | 270 | 10.4 |
| | 410 | 10.2 |
| | 500 | 9.99 |
| | 600 | 9.84 |
| | 700 | 9.55 |
| Cu-10 per cent $Al_2O_3$ | 85 | 12.0 |
| | 210 | 11.7 |
| | 400 | 11.3 |
| | 500 | 11.1 |
| | 640 | 10.7 |
| | 800 | 10.3 |

TABLE II.—*Continued.*

| Material | Temperature, deg Fahr | Modulus of Elasticity, *E*, psi |
|---|---|---|
| METAL - METAL OXIDE ALLOYS—*Continued* | | |
| Cu-1 per cent $SiO_2$ | 80 | 18.0 |
| | 210 | 17.5 |
| | 420 | 17.0 |
| | 500 | 16.7 |
| | 600 | 16.4 |
| | 700 | 16.0 |
| Cu-3 per cent $SiO_2$ | 80 | 17.4 |
| | 230 | 17.1 |
| | 400 | 16.7 |
| | 600 | 16.1 |
| | 800 | 15.5 |
| Cu-10 per cent $SiO_2$ | 85 | 14.7 |
| | 225 | 14.3 |
| | 405 | 14.0 |
| | 500 | 13.8 |
| | 800 | 12.8 |
| Cu-0 per cent $Cu_2O$ | 85 | 19.2 |
| | 230 | 18.6 |
| | 450 | 18.0 |
| | 600 | 17.3 |
| Cu-1 per cent $Cu_2O$ | 80 | $19.0 \times 10^6$ |
| | 200 | 18.7 |
| | 400 | 18.5 |
| | 600 | 17.9 |
| METAL - METAL OXIDE ALLOYS—*Continued* | | |
| Cu-3 per cent $Cu_2O$ | 80 | 14.4 |
| | 240 | 14.2 |
| | 400 | 13.9 |
| | 600 | 13.2 |
| | 800 | 12.0 |
| Ni-12 per cent $Al_2O_3$ | 80 | 26.3 |
| | 500 | 24.5 |
| | 600 | 24.1 |
| | 1000 | 21.9 |
| | 1200 | 20.8 |
| | 1500 | 18.8 |
| Ni-25 per cent $Al_2O_3$ | 80 | 26.3 |
| | 500 | 24.2 |
| | 1000 | 21.3 |
| | 1500 | 18.7 |
| | 1600 | 17.8 |
| SINTERED AND EXTRUDED | | |
| Pure nickel | 80 | 29.7 |
| | 600 | 28.2 |
| OTHERS[a] | | |
| Cu standard (OFHC) | 80 | 18.0<br>18.9 (**4**) |
| Al standard (2S) | 80 | 9.8<br>10.0 (**4**) |
| W standard | 80 | 58.7<br>60.0 (**4**) |

[a] Includes comparison with data of Köster (**4**).

TABLE III.—RESULTS OF THERMAL EXPANSION CORRECTION ON MODULUS DATA FOR ALLOY M-252.

| Temperature, deg Fahr | Modulus of Elasticity, psi | | | Error, per cent |
|---|---|---|---|---|
| | Uncorrected | Corrected[a] | Error† | |
| 80 | $31.00 \times 10^6$ | ... | ... | ... |
| 425 | 29.59 | $29.49 \times 10^6$ | $0.10 \times 10^6$ | 0.3 |
| 800 | 27.86 | 27.66 | 0.20 | 0.7 |
| 1200 | 25.66 | 25.36 | 0.30 | 1.2 |
| 1500 | 23.76 | 23.43 | 0.33 | 1.4 |
| 1600 | 22.89 | 22.56 | 0.33 | 1.5 |

† Approximate $\Delta$ error + 0.1 per cent per 100 F.

[a] Assumed linear coefficient of expansion = 0.00001 per deg Fahr.

small furnace in which holes were present for the suspension wires. The procedure was as follows:

1. The modulus was measured outside the furnace at room temperature. At this time the mode of vibration was checked.
2. The specimen was then placed in the furnace and rechecked at room temperature before making the rest of the measurements.
3. Modulus determinations were made as the specimen was heated. Testing was completed in less than 2 hr. The specimen was rechecked at room temperature after cooling.

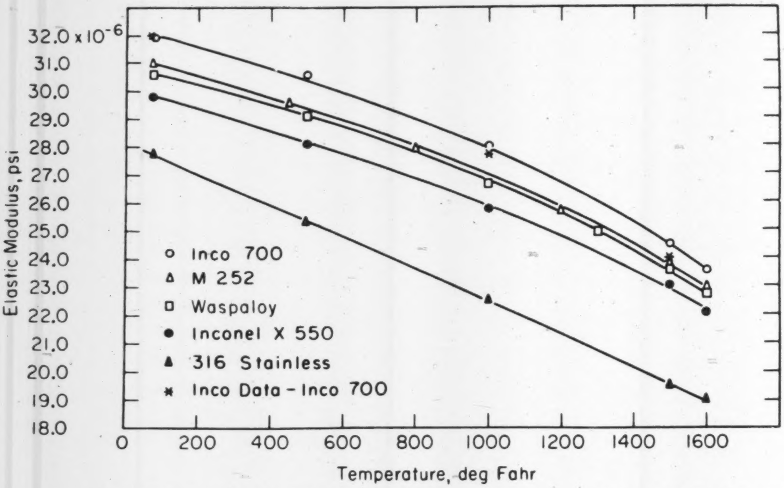

FIG. 2.—Elastic Modulus of Nickel-Base Heat-Resistant Alloys and Type 316 Stainless Steel.

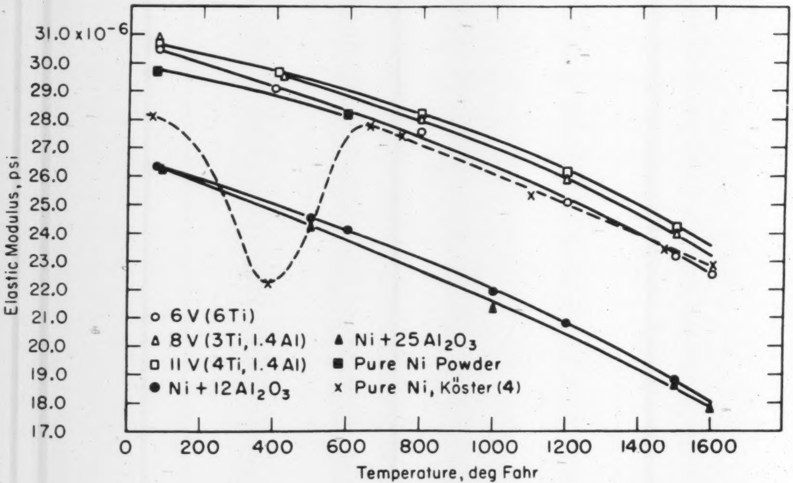

FIG. 3.—Elastic Modulus of Several Simple Vacuum Melted Ni-Cr-Ti-Al Alloys and for Pure Nickel and Two Nickel-Aluminum Oxide Alloys.

## TEST RESULTS

The data for all tests are presented in Table II, which lists the uncorrected modulus. Table III gives the results of a thermal expansion error calculation on one alloy, M-252.

Figures 2 to 6 present the modulus curves. Published data are included for comparison whenever such data could be found in the literature.

## DISCUSSION

It is believed that the data presented are for the true unrelaxed elastic modulus. There was not sufficient time for either thermal or atomic diffusion as mechanisms for relaxation to occur since

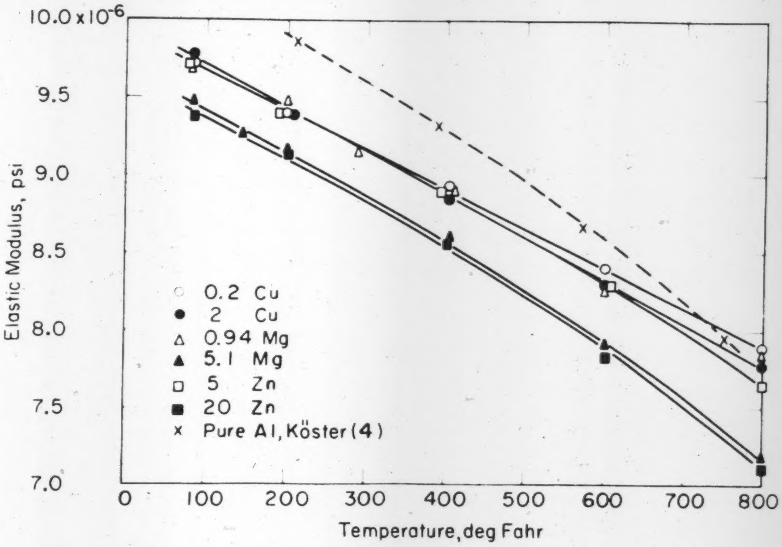

FIG. 4.—Elastic Modulus of Pure Aluminum and High Purity Alloys.

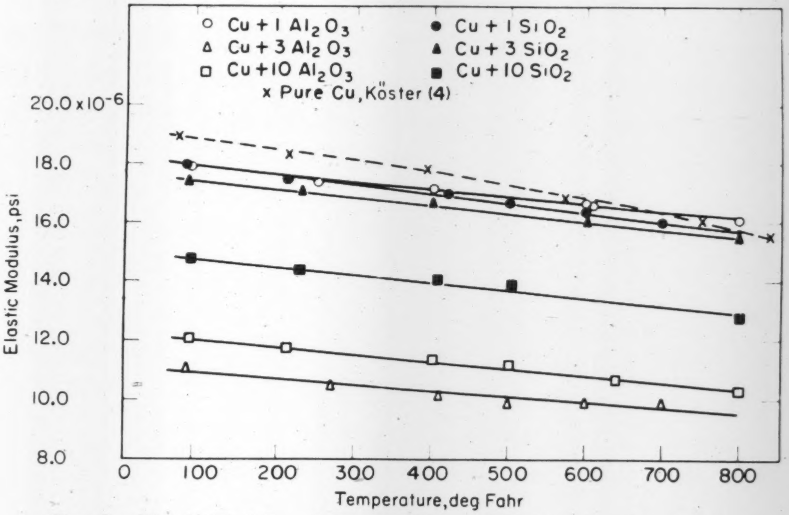

FIG. 5.—Elastic Modulus of Copper-Silica and Copper-Alumina Alloys.

the testing frequencies were in the range 3000 to 5000 cps.

At all temperatures the commercial nickel-base heat-resistant alloys show a higher elastic modulus than type 316 stainless steel. However, the curves are quite parallel (see Fig. 2). At temperatures of about 1400 to 1600 F, for these alloys, the stress for 100-hr rupture life (5) increases with increasing relative values of the elastic modulus, in an approximate way.

Figure 3 shows the elastic modulus data for three experimental vacuum melted alloys. These values for the relatively simple alloys are equal to and overlap the values for the more complex commercial alloys. In comparison to pure nickel the alloys all show equal or superior modulus values. The values obtained in the program on sintered extruded pure nickel agree quite well with the higher temperature values for nickel reported by Köster.

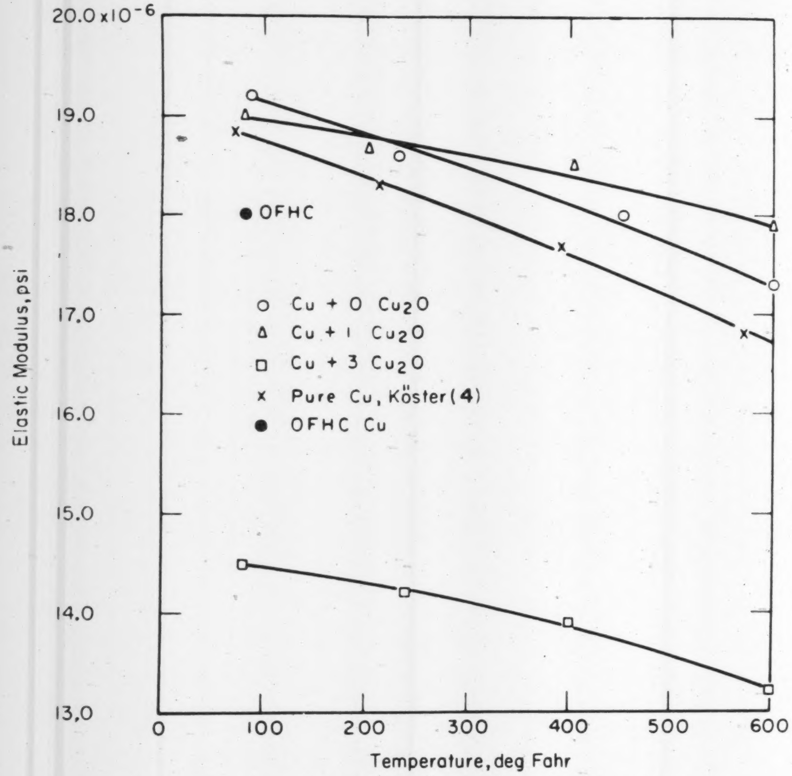

FIG. 6.—Elastic Modulus of Copper-Copper Oxide Alloys.

The dip in the pure nickel curve obtained by Köster (4) below 650 F is due to a magnetic effect, and was not observed in the data from the sintered extruded pure nickel. Köster observed that a magnetically saturated specimen exhibits a normal elastic modulus-temperature relationship, but when un-magnetized, the dip indicated in Fig. 3 occurs.

By comparison, the nickel-aluminum oxide alloys show parallel modulus curves but at much lower values; in fact, the values are more nearly equal to those of type 316 stainless steel (see Fig. 2).

Figure 4 shows the elastic modulus curves for pure aluminum (4) and for the high-purity aluminum alloys (6). Contrary to the results with the nickel alloys, pure aluminum shows the highest elastic modulus values for all temperatures up to about 700 F. Furthermore, with increasing alloy content the elastic modulus curve falls to lower values for all three alloy systems. Passing from a single-phase to a two-phase field does not have a noticeable effect on the modulus values. Unlike the nickel-base alloys, the strongest aluminum alloy (5 per cent magnesium) and one of the weakest (20 per cent zinc), in terms of rupture life at 500 and 700 F, show the lowest elastic modulus values.

Figures 5 and 6 show the elastic modulus curves for sintered extruded copper with $Cu_2O$, copper with $SiO_2$, and copper with $Al_2O_3$ alloys compared to pure copper. In Fig. 5 it will be noted that with increasing oxide content the elastic modulus values decrease, the decrease being somewhat larger for the $Al_2O_3$-containing alloys than for the $SiO_2$-containing alloys. Beyond an addition of about 1 volume per cent of oxide, the loss of elastic modulus can be large. Figure 6, on the other hand, shows an improvement in elastic modulus over pure copper for a 1 per cent $Cu_2O$ (by volume) addition. With 3 per cent $Cu_2O$ there is a large decrease in elastic modulus.

The improvements in high-temperature strength due to oxide additions in sintered extruded alloys of the type shown here are at the expense of the elastic modulus. The mechanism or cause of this decrease in elastic modulus is not known. It was noted, however, that damping effects were quite large in some of the copper alloys and prevented measurements at higher temperatures than those listed in Table II.

Although the thermal expansion correction is appreciable, a more refined apparatus would be necessary before making this correction to all measurements, and more precise knowledge of the thermal expansion coefficients would be required.

*Acknowledgments:*

The authors wish to thank those whose cooperation and help made this work possible; Mr. F. M. Young of the Acoustics Laboratory; Richard Charles of the Metallurgy Department, and the Atomic Instrument Corporation of Cambridge. Thanks are also due the Bureau of Aeronautics and Wright Air Development Center for support of programs which made possible these measurements.

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## REFERENCES

(1) F. Förster and W. Köster, "A New Method of Measurement for Young's Modulus and Damping," Abstract-Translation from *Zeitschrift für Metallkunde*, (Henry Brutcher, Translation No. 460), Vol. 29, pp. 109–123 (1937).

(2) M. H. Roberts and J. Nortcliffe, "Measurement of Young's Modulus at High Temperatures," *Journal*, Iron and Steel Inst., November, 1947, pp. 345–348.

(3) C. W. Andrews, "Effect of Temperature on the Modulus of Elasticity," *Metal Progress*, July, 1950, pp. 85–89, 96–100.

(4) W. Köster, "Die Temperaturabhangigkeit des Elastizitatsmoduls Reiner Metalle," *Zeitschrift für Metallkunde*, Vol. 39, pp. 1–16 (1948).

(5) R. F. Wilde, "Aging in Some Commercial Ni-Cr-Ti-Al Base Alloys," M. S. Thesis, Department of Metallurgy, Massachusetts Institute of Technology, December, 1955.

(6) Gordon D. Gemmell, "Effects of Solid Solution Alloying on Creep Deformation of Aluminum," Sc.D. Thesis, Department of Metallurgy, Massachusetts Institute of Technology, January, 1956.

# DISCUSSION

Mr. J. G. Kaufman[1] (*presented in written form*).—The authors have included some interesting data on aluminum alloys in their paper on the determination of dynamic elastic moduli. Their room temperature data are in reasonable agreement with dynamic modulus values obtained at the Alcoa Research Laboratories by an entirely different method. This agreement raises some questions, however.

Alcoa's dynamic modulus values were determined from simple vibrations of cantilever beams, and the tests were restricted primarily to aluminum-copper alloys. Repeatedly the moduli for these alloys were found to be 9.6 to 9.9 × $10^6$ psi. This did not appear compatible with values around 10.6 × $10^6$ psi determined from static tension tests. The authors have now presented data in general agreement with the lower values, also obtained by a dynamic vibration method. It is not clear to us why dynamically determined room temperature elastic moduli would be lower than statically determined values in which some time-dependent strain could be involved.

Such time-dependent effects become more obvious at the elevated temperatures, where static moduli drop much faster than dynamic moduli, particularly above 400 F. Whereas static modulus values of aluminum-copper alloys, for example (not high purity), determined at Alcoa[2] drop to about 73 per cent of the room temperature values at 600 F, dynamic values as shown by the authors are still around 85 to 87 per cent of their room temperature values. This is even more pronounced in the aluminum-zinc systems where at 600 F static and dynamic moduli are about 50 and about 84 per cent, respectively, of their room temperature values. Since such differences do exist, and have been reported before,[3] it is clear that consideration must be given when choosing moduli for a specific design problem as to whether the statically or dynamically determined values are most applicable.

One other point seems worth noting and once again it is a contrast between static and dynamic modulus values. It is noted from Fig. 4 that unalloyed aluminum appears to have a higher dynamic modulus than do aluminum alloys and, in fact, as alloying constituent increases, modulus decreases. This is contrary to results obtained from static tension tests in which pure aluminum seems to have about the lowest elastic modulus (about 10.0 × $10^6$) and as alloying constituents are added, the modulus rises. Considering commercial alloys,[4] modulus values go up to about

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[1] Research Engineer, Mechanical Testing Division, Alcoa Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] E. C. Hartmann, F. M. Howell, and R. L. Templin, "How to Use High Strength Aluminum Alloy," *Aviation Week*, Oct. 10, 1949.

[3] W. S. Hyler and H. J. Grover, "Materials-Property-Design Criteria for Metals," *Technical Report 55-150*, Wright Air Development Center, Nov., 1955.

[4] "Alcoa Aluminum Handbook," Aluminum Company of America (1956).

10.8 × $10^6$ psi in the aluminum-copper series, 10.4 × $10^6$ psi in the aluminum-magnesium and aluminum-zinc series. This type of behavior is generally more consistent with estimates of modulus values computed by weighted averages based on chemical composition, which in the majority of cases provide good values for aluminum alloys. In such computations, those alloys with constituents with moduli above that of pure aluminum would have moduli that increase as alloying constituent increases.

It does not seem reasonable that the only reason for the difference in static and dynamic behaviors could be the fact that the authors used high-purity binary alloys, whereas commercial alloys naturally have some impurities plus minor alloying constituents. We wonder if the authors have considered such differences at all or made any attempt to compare their dynamic moduli with static moduli on the same or similar alloys.

MESSRS. R. F. WILDE AND N. J. GRANT (*authors*).—We thank Mr. Kaufman for his comments on the differences between dynamically and statically measured elastic moduli in aluminum alloys.

Mr. Kaufman's observations are paradoxical because, all other things being equal, one expects the dynamic modulus to be equal to or greater than the static modulus. Other investigators[5] have found that the room temperature elastic modulus as measured on similar specimens by both methods were consistent and within the limits of experimental error.

The use of identical specimens may be important in obtaining consistency. Aluminum does not exhibit the same degree of anisotropy as some other face-centered cubic materials, but it is possible that some of the differences observed can be attributed to anisotropy effects. These may be more pronounced if alloying increases anisotropy.

Although the authors did not measure static values, a determination on pure (99.99 per cent) aluminum resulted in a dynamic modulus of 9.8 × $10^6$ psi, consistent with published static values, but lower than that reported by Köster[6] and shown in Fig 4.

MR. R. E. PHILLEO.[7]—What type of transducers were used and what was the coupling between the transducer and the specimen?

MESSRS. WILDE AND GRANT.—In this work, the transducers were rather primitive. The driver consisted of a permanent magnet speaker with the cone removed and a rigid wire support fastened to the spider. A thin nichrome wire was hooked to the support and a loop was made at the bottom, from which one end of the specimen was supported at its nodal point. The receiver was a high output phonograph crystal, from which another wire supported the specimen at the other nodal point.

It is necessary to isolate the system from external vibration. More refined apparatus has extended the upper temperature limit to 2000 F in later work.

A MEMBER.—I think it is generally conceded that dynamic moduli are higher than those determined statically at elevated temperatures.

I wonder if the authors of the paper can give any advice as to just what point one should begin using moduli determined by dynamic methods rather than static methods for any of the materials that were discussed in the paper.

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[5] R. S. Mateer and J. B. Hess, "Development of Aluminum Base Alloys by Techniques Involving Powder Metallurgy and Conventional Metal Working Methods," *Project Report*, No. MS PR 54-27, Kaiser Aluminum and Chemical Corp., June 14, 1954.

[6] W. Köster, "Die Temperaturabhangigkeit des Elastizitätsmodulus reiner Metalle," *Zeitschrift für Metallkunde*, Vol. 39, pp. 1–16 (1948).

[7] Research Engineer, Fire Research Section, Portland Cement Assn., Chicago, Ill.

MESSRS. WILDE AND GRANT.—There are several considerations on the use of dynamic moduli:

1. The dynamic modulus curve for a given material becomes significantly higher at temperatures and strain rates where the material exhibits high temperature behavior. This is above 1400 F for a nickel base super alloy, while it is only 400–500 F for an aluminum base alloy.

2. Any application where loading involves a high strain rate, such as flutter, and where the principal stresses are alternating or impulsive is a good one to consider using the dynamic modulus.

3. Design criteria where static stresses are paramount indicate the use of static moduli.

MR. HERBERT T. CORTEN.[8]—What magnitude of strain was involved in the tests? Were these small elastic strains or larger strains associated with, say, the yield strength?

MR. WILDE.—The strains were so small as to be infinitesimal. We did not measure them but the original work performed by Föster and Köster[6] showed an amplitude of 30 to 5000 $\mu\mu$. That apparently is their magnitude here. Sonic vibration of a bar does not require a large strain.

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[8] Associate Professor, Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

# EFFECT OF COMPOSITION ON THE ELEVATED-TEMPERATURE PROPERTIES OF DUCTILE IRON*

By R. D. Schelleng[1] and J. T. Eash[1]

## Synopsis

Creep properties of several ferritic ductile irons at temperatures from 600 to 1000 F are presented and the effects of variations in phosphorus, copper, and molybdenum contents are shown. Stress-rupture data at 800 and 1000 F and tensile strength data from room temperature to 1000 F are included. Creep and rupture data on several austenitic ductile irons containing 20 to 35 per cent nickel, 2 to 3 per cent chromium, and 0 to 1 per cent molybdenum are also presented. A wide range of properties at elevated temperatures are obtainable in ductile iron through alloy additions. The properties of the ferritic material are comparable to those of low-carbon steels, and those of the nickel-austenitic materials are similar to the properties of some stainless steels.

Ductile iron, because of its good castability, strength, and resistance to growth, holds considerable promise for use at elevated temperatures. Unlike the strength of gray iron, which is controlled largely by the graphite flake size and distribution, the properties of ductile iron are controlled mainly by the properties of the matrix. Thus, the strength of ductile iron can be influenced considerably by alloy additions.

Many writers have explored the variations in room temperature properties of ductile irons of different compositions. However, the information on the elevated temperature properties of ductile iron is quite limited. Saunders and Sinnott (**1**)[2] reported the properties of a single melt of partially pearlitic ductile iron at temperatures up to 1200 F. Wilks, Matthews, and Kraft (**2**) published tensile, creep, and stress-rupture data on heats of ferritic, pearlitic, and nickel-austenitic ductile irons tested at 800 to 1200 F and Sefing (**3**) recently presented limited data on the tensile and rupture strengths of some nickel-austenitic irons at temperatures from 1000 to 1400 F. Foley (**4**) has published a compilation of creep and rupture data on ferritic ductile iron from several sources, including two of the above references.

This paper discusses the effects of variations in phosphorus, copper, and molybdenum contents and heat treatment on the creep resistance of ferritic and pearlitic ductile iron at 800 F. Additional data on the tensile strength, stress-rupture, and creep properties of a nickel-copper, ductile iron at temperatures up to 1000 F and of nickel-austenitic ductile iron at temperatures up to 1200 to 1400 F are also presented.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p 945.

The materials used in this investigation were melted in an indirect-arc rocking furnace lined with a neutral refractory. The furnace charges consisted of pig iron, steel scrap, electrolytic nickel, and ferro alloys. The iron was reladled at 2700 F onto nickel-magnesium alloy and then 0.6 per cent ferro-85-silicon was added to the metal stream during a second reladling operation for graphite inoculation. The metal was poured into double keel bar molds, $\frac{5}{8}$ in. by $1\frac{5}{8}$ in. by 16 in., and 1 in. by 1 in. by 7 in. Creep test specimens were machined from the $\frac{5}{8}$-in. bars and tensile and stress-rupture specimens were obtained from the 1-in. bars. The creep testing procedures used at the Bayonne Laboratory of The International Nickel Co. are adequately described in the literature (**5**) and in general follow ASTM Recommended Practice E 22 – 41. The tensile and stress-rupture testing procedures also comply with ASTM Recommended Practices E 21 – 43 and E 85 – 50 T respectively.

## FERRITIC DUCTILE IRONS

Soon after the commercial development of the spheroidal graphite irons began, there was a need for data on high-temperature properties, particularly regarding creep. Initial tests were performed on an iron of conventional composition and of low alloying element content. The results presented in Fig. 1 show that the annealed iron had a creep rate of 1 cru[3] at 800 F under a stress of 8500 psi; and in the as-cast stress-relieved condition, it withstood a stress of 11,500 psi. Photomicrographs of this iron, Figs. 2 and 3, indicate the presence of a greater amount of pearlite in the stress-relieved specimen which is responsible for its greater strength.

These results, surprisingly, were similar to the creep strength of gray iron in spite of the much higher room-temperature properties possessed by ductile iron. Also, reports from the field had indicated that ductile iron of supposedly normal composition would have superior creep resistance. It was obvious that an in-

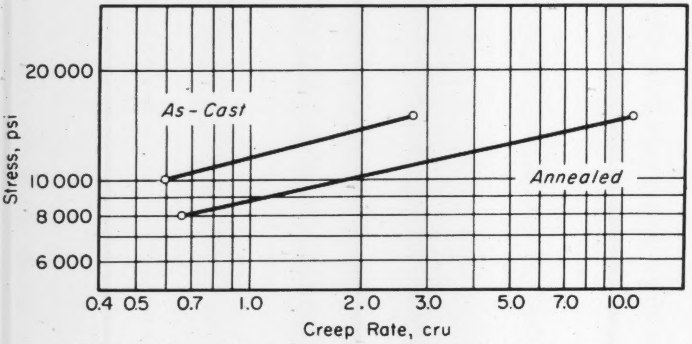

FIG. 1.—Creep Properties of Ductile Iron No. 1 at 800 F.
As-cast material was stress relieved at 900 F for 5 hr.
1 creep rate unit = 0.0001 per cent creep per hr.

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[3] 1 cru = 1 creep rate unit = 0.0001 per cent creep per hr.

vestigation of the metallurgical factors affecting high-temperature properties was necessary.

*Effect of Phosphorus:*

Normally the amount of phosphorus in ductile iron is minimized because it decreases the toughness of the material. However when present in quantities of 0.05 to 0.09 per cent impairment of the ductility is not excessive and the tensile strength of the iron is considerably higher than shown by irons essentially free of phosphorus. The present work has shown that by maintaining the phosphorus content of ductile iron at around 0.09 per cent a significant improvement in creep resistance is secured. Figure 4 presents data obtained from tests made at 800 F and 20,000 psi stress on irons with phosphorus contents varying from 0.017 to 0.097 per cent. The specimens tested in the annealed condition exhibited rapid creep, but a definite superiority of the irons with the greater phosphorus contents was observed. In the as-cast condition, stress relieved for 5 hr at 900 F, these irons were much stronger because of the greater amounts of pearlite present; a minimum creep rate of only 0.63 cru was obtained in the iron containing 0.085 per cent phosphorus.

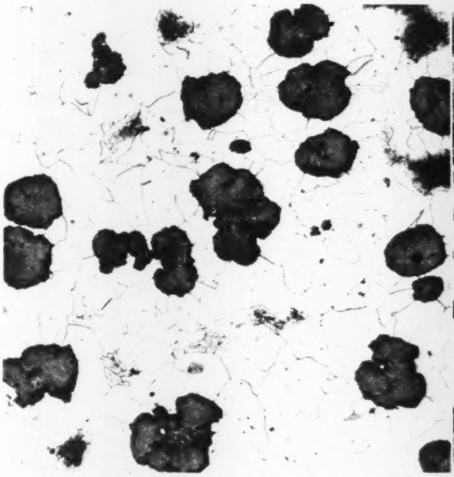

FIG. 2.—Microstructure of Annealed Ductile Iron No. 1, Nital Etched (×100).

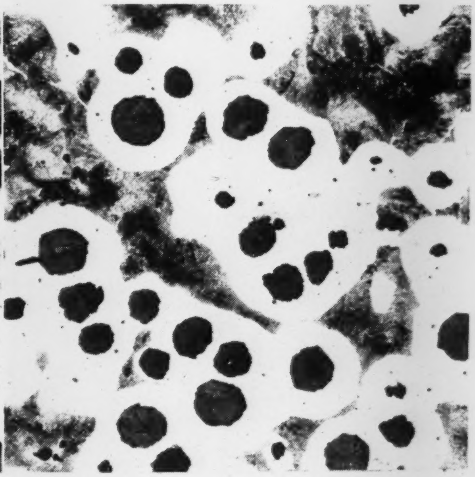

FIG. 3.—Microstructure of As-Cast Ductile Iron No. 1, Picral Etched (×100).

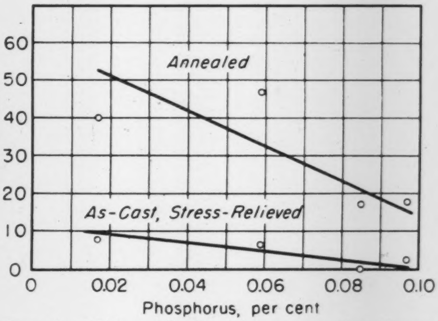

FIG. 4.—Effect of Phosphorus on the Creep Rate of Ductile Iron Stressed at 20,000 psi at 800 F. Stress Relief was Performed at 900 F for 5 hr.

*Effect of Molybdenum:*

Molybdenum was found to be very effective for increasing the creep resistance of spheroidal graphite iron. Figure 5 presents the results obtained on two compositions made from low-phosphorus base irons and containing 1 per cent nickel with 0.24 and 0.81 per cent molybdenum.

At 800 F, the 0.8 per cent molybdenum iron in the annealed condition showed a minimum creep rate of 0.1 cru under a stress of 22,000 psi and a 1 cru rate at 28,500 psi; however, third stage creep started after about 1600 hr with a stress of 25,000 psi, which indicated the safe working load to be somewhat less where stable long-time service would be required. At 1000 F, a minimum creep rate of 1 cru was produced at a stress of 8000 psi. The microstructure of the annealed iron, Fig. 6, had a fine-grained ferritic matrix containing a small amount of pearlite and dispersed carbides in the cell boundaries.

In the as-cast, stress-relieved condition, the matrix of the 0.8 per cent molybdenum iron contained a mixture of ferrite, pearlite, and bainite, Fig. 7. When tested at 800 F, this specimen showed a creep rate of 0.08 cru at a stress of 20,000 psi, which is of the same order of magnitude as found for the material in the annealed condition. However, the pearlitic-bainitic as-cast material would not be expected to enter third stage creep as soon as the ferritic, annealed material.

The iron containing 0.24 per cent molybdenum was less resistant to creep than the iron having 0.8 per cent molybdenum as shown in Fig. 5; however, its creep strength was twice that of the low-alloy samples.

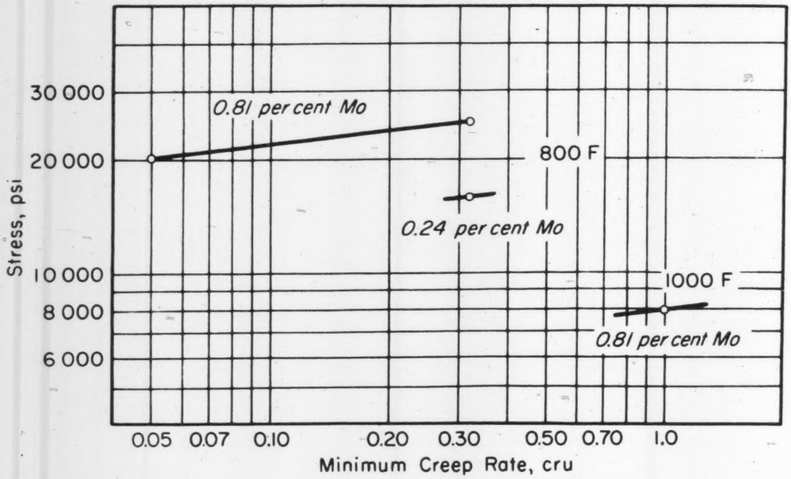

FIG. 5.—Creep of Annealed Ductile Iron Containing Molybdenum, Alloys 3 and 4.

The amount of molybdenum which can be added to ductile iron is limited, because this alloying element tends to form carbides and excessive amounts of it will embrittle the material. The iron to which 0.81 per cent molybdenum had been added contained an appreciable amount of fine, intercellular carbide. This carbide is quite stable, as demonstrated by the fact that the annealing treatment at 1750 F for 16 hr did not completely decompose it. The iron which contained 0.24 per cent molybdenum had only a slight amount of this stable carbide.

*Effect of Copper:*

Copper has been added frequently to ductile iron, mainly as part of the addition agent for magnesium. It tends to stabilize the pearlite and thereby lowers the ductility and toughness. It also raises the impact transition temperature; consequently its use has been reduced in recent years. Copper does have a beneficial effect on creep resistance, however, and when used in combination with nickel some of its detrimental effects are overcome.

Creep data obtained on ductile irons containing about 1.1 per cent nickel, 0.6 per cent copper and 0.085 per cent phosphorus are presented in Fig. 8. In the annealed condition, the material showed a minimum creep rate of 1 cru at 800 F when stressed at 20,500 psi, which is an improvement $2\frac{1}{2}$-fold over the low-alloyed irons and approaches the strength of the iron containing 0.8 per cent molybdenum.

Since the alloy cost of the nickel-copper iron would be relatively low compared to the nickel-molybdenum iron, considerable attention was given to investigation of the former.

It will be noted from the microstructure of the copper-bearing ductile iron shown in Fig. 9 that an appreciable amount of pearlite is present after the annealing treatment. The heat treatment employed was as follows: heated at 1650 F for 2 hr, furnace cooled to 1275 F, held 5 hr, and air cooled. This heat treatment will produce a ferritic matrix in irons of conventional composition. The high manganese and copper contents in this iron tend to stabilize the pearlite. The ferrite grain size of the iron after this anneal was ASTM 7.[4] When annealed at 1750 F for 16 hr, furnace cooled to 1500 F, then cooled at 10 F per hr to 1250 F followed by air cooling, a completely ferritic matrix with a fer-

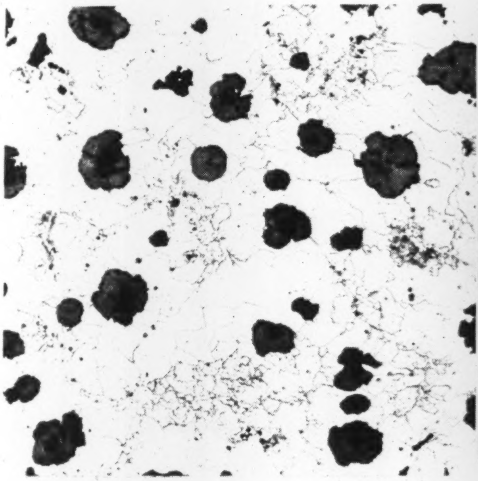

Fig. 6.—Microstructure of Annealed Ductile Iron Containing 0.81 per cent Molybdenum, Alloy No. 3, Nital Etched (×100).

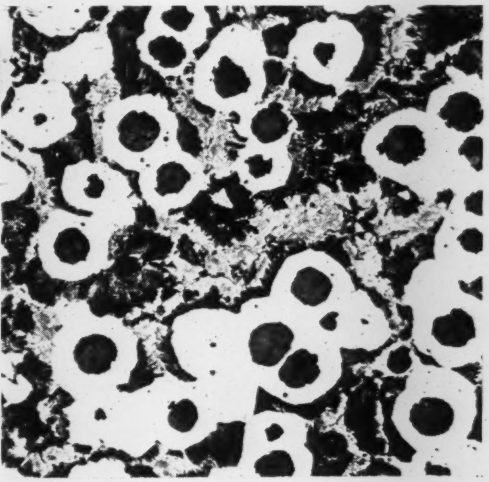

Fig. 7.—Microstructure of As-Cast Ductile Iron Containing 0.81 per cent Molybdenum, Alloy No. 3, Picral Etched (×100).

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[4] Methods for Estimating the Average Ferrite Grain Size of Low-Carbon Steels (E 89 – 52), 1955 Book of ASTM Standards, Part 1, p. 1690.

rite grain size of ASTM 5 was produced (Fig. 10). In this condition, this iron showed a creep rate of only 0.25 cru at 800 F under 20,000 psi stress which is a marked improvement in creep resistance in spite of the decomposition of the pearlitic matrix and coarsening of the ferrite grain size.

An as-cast, stress-relieved specimen of this copper-bearing iron exhibited creep properties at 800 F similar to those shown by the material given the long annealing treatment described above. However, the stress-relieved specimen was superior to the annealed in that it had not entered third stage creep after 5650 hr of testing. The creep rate of the annealed material started to increase slowly from its minimum value after 3500 hr.

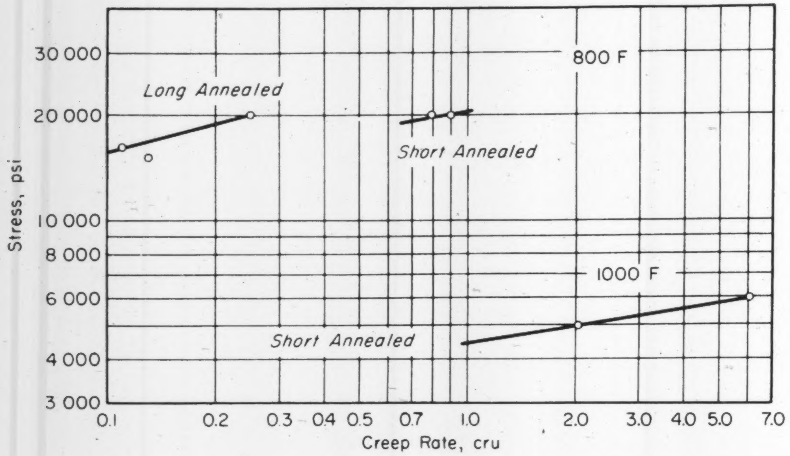

Fig. 8.—Creep Properties of Nickel-Copper Ductile Iron, Alloy No. 5.

The creep properties of this nickel-copper ductile iron at 600 and 1000 F were also explored. Two bars of the material were given the shorter annealing treatment and were tested with 30,000 and 40,000 psi stress at 600 F. A small amount of first stage creep occurred, but after about 1800 hr the creep rates were essentially zero. At this temperature yield strengths become more significant than creep.

At 1000 F the stress for a 1 cru creep rate in the nickel-copper iron after the short anneal was found to be 4400 psi. In the as-cast, stress-relieved condition the material was much weaker (Table I), which indicates that a high temperature heat treatment is essential for optimum creep resistance in copper containing ductile irons.

*Stress Rupture Tests.*—The results of stress rupture tests on the annealed nickel-copper ductile iron are presented in Fig. 11. These data indicate that the stress to produce rupture in 1000 hr for this material should be about 27,000 psi at 800 F and 7500 psi at 1000 F. At 800 F, the strength of this material is greater than the average value for low-alloyed ductile iron reported by Foley (**4**). At 1000 F, however, the strength of the nickel-copper iron is about the same as the average indicated by the latter.

*Precipitation Hardening.*—The precipitation hardening characteristics of copper-bearing iron-carbon alloys are well known. Mild precipitation hardening of the nickel-copper ductile iron occurred during the creep run at 800 F as is shown by the following data:

| | Before Creep Test | After Creep Test |
|---|---|---|
| Brinell Hardness | 172 | 185 |
| Tensile Strength, psi | 77 500 | 88 000 |
| Yield Strength, psi | 52 000 | 79 500 |
| Elongation in 2 in., per cent | 16 | 8 |

It is important to note that the iron maintained a high degree of ductility and did not become embrittled by the long exposure.

Tests were made to evaluate the effect of the precipitation hardening reaction on the rupture life of this iron. Tests were run on material which had been given a solution treatment at 1350 F for 2 hr after an annealing treatment, which produced a completely ferritic matrix. The results of these tests are shown below:

| Heat Treatment | Temperature, deg Fahr | Stress, psi | Life, hr | Elongation in 2 in., per cent |
|---|---|---|---|---|
| Annealed[a] | 800 | 44 000 | 10.6 | 8 |
| Annealed and solution treated[b] | 800 | 44 000 | 33.2 | 8 |
| Annealed | 1000 | 15 000 | 45.4 | 7 |
| Annealed and solution treated | 1000 | 15 000 | 26.1 | 5 |

[a] Heated at 1650 F for 2 hr, furnace cooled to 1500 F, cooled at 10 F per hr to 1250 F, and air cooled.

[b] Annealed as above, reheated to 1350 F, held 2 hr, air cooled.

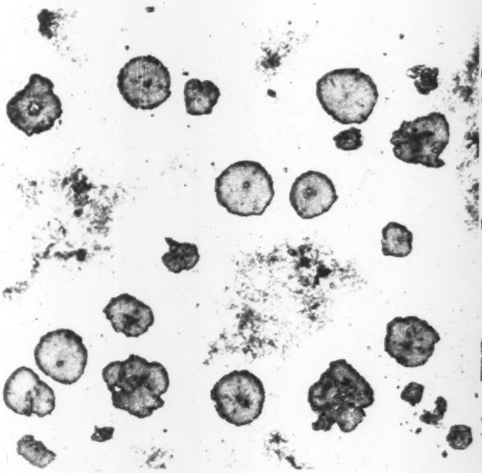

Fig. 9.—Microstructure of Nickel-Copper Ductile Iron Alloy No. 5 Annealed at 1650 F, Nital Etched (×100).

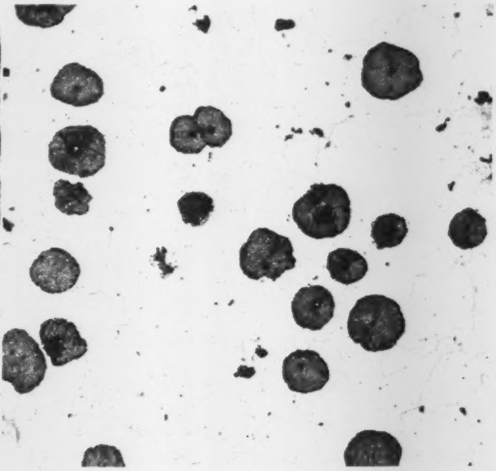

Fig. 10.—Microstructure of Nickel-Copper Ductile Iron Alloy No. 5 Given Long Anneal at 1750 F, Nital Etched (×100).

The life of the solution-treated specimen tested at 800 F was over three times that of the annealed specimen with no sacrifice in elongation. The microstructures of these irons showed that the precipitate in the solution-treated specimen, Figure 12, was more abundant and uniformly distributed than that in the annealed specimen. It is thought that a similar more-favorable distribution of the precipitate is responsible for the superior creep strength at 800 F of the coarse-

TABLE I.—SUMMARY OF CREEP AND STRESS-RUPTURE DATA.

| Alloy | Composition, per cent | | | | | | | | | Condition | Matrix | Temperature, deg Fahr | Stress for 1 cru,[d] psi | Stress for 0.1 cru, psi | Stress for 1000 hr Rupture, psi |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Total Carbon | Silicon | Manganese | Nickel | Copper | Chromium | Molybdenum | Phosphorus | Magnesium | | | | | | |
| No. 1 | 3.54 | 2.27 | 0.40 | 0.56 | 0.15 | ... | ... | 0.017 | 0.050 | Annealed[a] | Ferrite | 800 | 8 500 | ... | ... |
| | 3.54 | 2.27 | 0.40 | 0.56 | 0.15 | ... | ... | 0.017 | 0.050 | As-cast, stress relieved[b] | Ferrite and pearlite | 800 | 11 500 | ... | ... |
| No. 2 | 3.49 | 2.50 | 0.37 | 1.22 | ... | ... | ... | 0.085 | 0.064 | Annealed | Ferrite | 800 | 12 000 | ... | ... |
| | 3.49 | 2.50 | 0.37 | 1.22 | ... | ... | ... | 0.085 | 0.064 | As-cast, stress relieved | Pearlite | 800 | 23 000 | ... | ... |
| No. 3 | 3.61 | 2.43 | 0.47 | 1.19 | ... | ... | 0.81 | ... | 0.050 | Annealed | Ferrite | 800 | ... | 22 000 | ... |
| | 3.61 | 2.43 | 0.47 | 1.19 | ... | ... | 0.81 | ... | 0.050 | As-cast, stress relieved | Ferrite, pearlite bainite | 800 | ... | 20 500 | ... |
| No. 4 | 3.59 | 2.43 | 0.40 | 1.08 | ... | ... | 0.24 | ... | 0.050 | Annealed | Ferrite | 800 | ... | 14 000 | ... |
| No. 5 | 3.49 | 2.46 | 0.78 | 1.08 | 0.56 | ... | ... | 0.086 | 0.072 | Annealed | Ferrite, pearlite | 800 | 20 500 | ... | 27 000 |
| | 3.49 | 2.46 | 0.78 | 1.08 | 0.56 | ... | ... | 0.086 | 0.072 | High temperature annealed[c] | Ferrite | 800 | ... | 15 000 | ... |
| | 3.49 | 2.46 | 0.78 | 1.08 | 0.56 | ... | ... | 0.086 | 0.072 | As-cast and stress relieved | Pearlite | 800 | ... | 16 000 | ... |
| | 3.49 | 2.46 | 0.78 | 1.08 | 0.56 | ... | ... | 0.086 | 0.072 | Annealed | Ferrite, pearlite | 1000 | 4 400 | ... | 7 500 |
| | 3.49 | 2.46 | 0.78 | 1.08 | 0.56 | ... | ... | 0.086 | 0.072 | As-cast, stress relieved | Pearlite | 1000 | 3 000 | ... | ... |
| No. 6 | 2.98 | 2.20 | 1.15 | 20.48 | ... | 2.36 | ... | ... | 0.085 | As-cast | Austenite, carbide | 1000 | 23 000 | 9 000 | 28 000 |
| | 2.98 | 2.20 | 1.15 | 20.48 | ... | 2.36 | ... | ... | 0.085 | As-cast | Austenite, carbide | 1200 | 8 000 | 3 400 | 12 000 |
| No. 7 | 2.73 | 2.30 | 0.86 | 20.56 | ... | 1.70 | 0.71 | ... | 0.10 | As cast | Austenite, carbide | 1100 | 16 000 | ... | 26 000 |
| | 2.73 | 2.30 | 0.86 | 20.56 | ... | 1.70 | 0.71 | ... | 0.10 | As-cast | Austenite, carbide | 1300 | 5 600 | ... | 10 000 |
| No. 8 | 2.37 | 1.89 | 0.62 | 30.83 | ... | 3.01 | ... | ... | 0.14 | As-cast | Austenite, carbide | 1100 | ... | ... | 23 500 |
| | 2.37 | 1.89 | 0.62 | 30.83 | ... | 3.01 | ... | ... | 0.14 | As-cast | Austenite, carbide | 1300 | ... | ... | 9 700 |
| No. 9 | 2.38 | 1.99 | 0.62 | 30.18 | ... | 3.05 | 0.95 | ... | 0.13 | As-cast | Austenite, carbide | 1100 | ... | ... | 30 500 |
| | 2.38 | 1.99 | 0.62 | 30.18 | ... | 3.05 | 0.95 | ... | 0.13 | As-cast | Austenite, carbide | 1300 | 7 000 | ... | 11 000 |
| No. 10 | 2.18 | 1.51 | 0.56 | 35.17 | ... | 2.85 | ... | ... | 0.12 | As-cast | Austenite, carbide | 1100 | ... | ... | 25 000 |
| | 2.18 | 1.51 | 0.56 | 35.17 | ... | 2.85 | ... | ... | 0.12 | As-cast | Austenite, carbide | 1300 | ... | ... | 10 000 |
| No. 11 | 2.24 | 1.48 | 0.58 | 35.21 | ... | 2.61 | 0.73 | ... | 0.12 | As-cast | Austenite, carbide | 1100 | ... | ... | 32 000 |
| | 2.24 | 1.48 | 0.58 | 35.21 | ... | 2.61 | 0.73 | ... | 0.12 | As-cast | Austenite carbide | 1300 | 6 000 | ... | 12 000 |
| No. 12 | 2.29 | 5.65 | 0.64 | 27.98 | ... | 4.60 | ... | ... | 0.12 | As-cast | Austenite, carbide | 1100 | ... | ... | 17 000 |
| | 2.29 | 5.65 | 0.64 | 27.98 | ... | 4.60 | ... | ... | 0.12 | As-cast | Austenite, carbide | 1300 | ... | ... | 6 200 |
| No. 13 | 2.19 | 5.66 | 0.58 | 27.37 | ... | 4.36 | 0.87 | ... | 0.091 | As-cast | Austenite, carbide | 1100 | ... | ... | 19 000 |
| | 2.19 | 5.66 | 0.58 | 27.37 | ... | 4.36 | 0.87 | ... | 0.091 | As-cast | Austenite, carbide | 1300 | ... | ... | 7 500 |

[a] 1650 F—2 hr, furnace cool to 1275 F, air cool.

[b] 900 F—5 hr, air cool.

[c] 1750 F—16 hr, cool 10 F per hr to 1250 F, air cool.

[d] Creep rate of 0.0001 per cent per hr.

grained, nickel-copper ductile iron annealed at 1750 F for 16 hr over the finer grained, partially pearlitic iron annealed at 1650 F for 2 hr. The residual pearlite in the latter material obscured the precipitate and prevented microscopic analysis. At 1000 F, the precipitate in the solution-treated stress-rupture specimen was localized in the grain boundaries (Fig. 13), and the rupture life was only half that of the annealed specimen with a decrease in elongation.

*Tensile Properties.*—The short-time elevated temperature tensile properties of the annealed nickel-copper iron are compared with a 1.35 per cent nickel iron in Fig. 14. As might be expected, the copper-bearing iron was somewhat stronger at all temperature levels. An ultimate strength of some 70,000 psi was maintained up to 600 F followed by a decrease to about 35,000 psi at 1000 F. The yield strength diminished more gradually. Usually low-alloy content ductile iron exhibits a minimum in its elongation at around 800 to 900 F; it is interesting to note that the lowest values of elongation for copper-containing iron were secured at 1000 F. The tensile values of the nickel iron are slightly higher than those reported by Wilks *et al* (2) and the average values reported by Foley (4); however they are of the same order of magnitude.

The strength of ferritic ductile iron compares quite favorably with that of annealed low-carbon steel. The yield strengths of both of the irons tested are superior to the values reported for both cast and wrought low-carbon steel (6,7), and the tensile values for the nickel-copper iron fall between those for cast and wrought low-carbon steel. The tensile values of the nickel-containing iron approximate those reported for wrought low-carbon steel except at room temperature where the iron is superior.

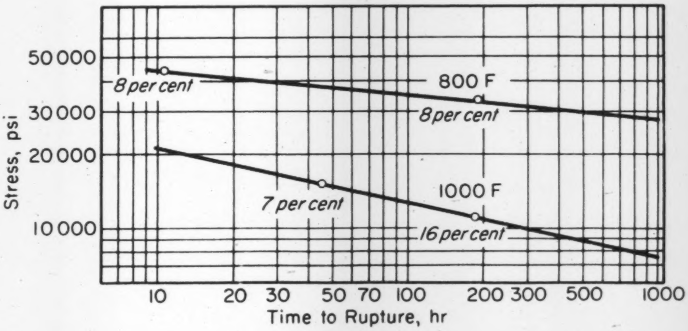

FIG. 11.—Stress-Rupture Data for Annealed Nickel-Copper Ductile Iron, Alloy No. 5.

### *Structural Stability:*

No microstructural changes were observed in the irons tested at 800 F. At 1000 F the pearlite gradually decomposed. The annealed copper-bearing iron initially contained rather large amounts of pearlite as shown in Fig. 9. After testing at 1000 F most of this pearlite had decomposed. Thus, the utilization of any improvement in strength which might be procured by using the material in a pearlitic condition at the lower temperatures would have to be in applications of close temperature control.

*Summary of Data on Ferritic Irons:*

A summary of the more important creep results on ferritic irons compared with published information on steels (6,7) is presented in Fig. 15. The superiority of the copper- and molybdenum-containing irons over the low-alloyed iron is evident. The strength of the copper-bearing material approximates that of low-carbon steel, and the properties of the iron containing 0.8 per cent molybdenum approach those of 0.5 per cent molybdenum steel. The data reported in Foley's compilation were obtained on irons of generally higher phosphorus and copper contents than the low-alloy iron of the present work, and one of the irons was partially pearlitic. These differences account for the higher values reported by Foley.

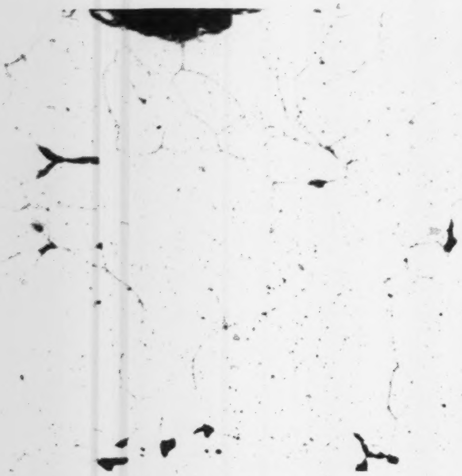

FIG. 12.—Microstructure of Annealed and Solution-Treated Nickel-Copper Ductile Iron Alloy No. 5 Stress-Rupture Tested at 800 F Showing Uniform Distribution of Copper-Rich Precipitates, Nital Etched (×500).

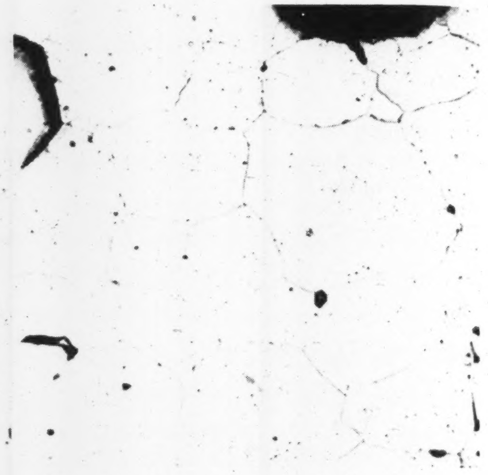

FIG. 13.—Microstructure of Annealed and Solution Treated Nickel-Copper Ductile Iron Alloy No. 5 Stress-Rupture Tested at 1000 F, Nital Etched (×500). Copper-Rich Precipitates are Concentrated in Grain Boundaries.

## AUSTENITIC DUCTILE CAST IRON

*Creep Tests:*

Creep data obtained on a group of austenitic ductile irons containing 20 to 35 per cent, nickel, 1.70 to 3 per cent chromium, and 0 to 1 per cent molybdenum over the temperature range of 1000 to 1300 F are shown in Figs. 16 and 17. These results show that increase

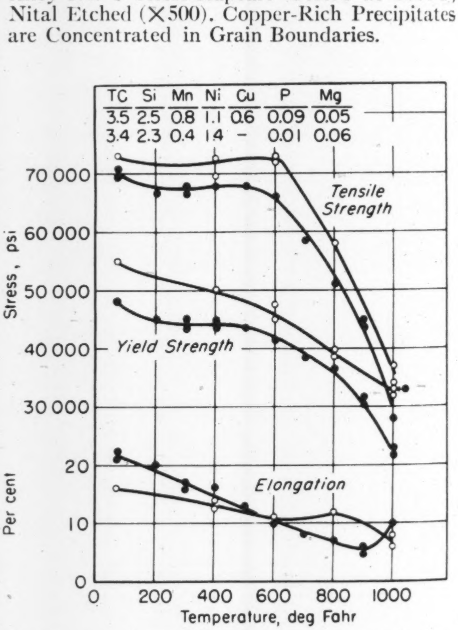

FIG. 14.—Tensile Properties of Ductile Iron at Elevated Temperatures.

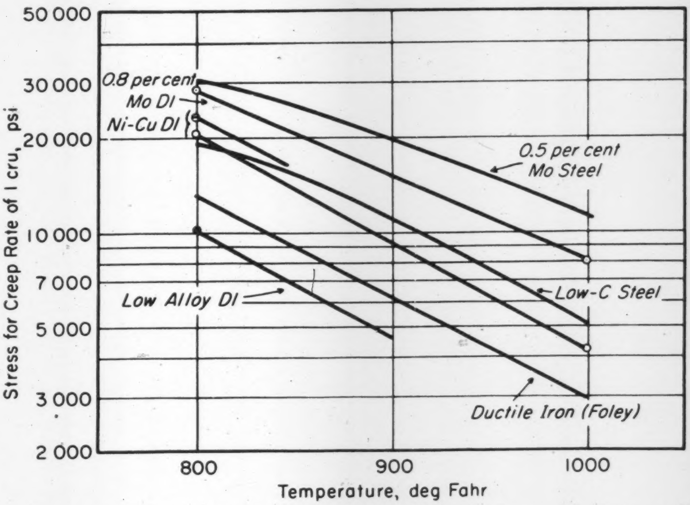

Fig. 15.—Comparison of the Creep Strength of Ductile Iron with Data on Steel.

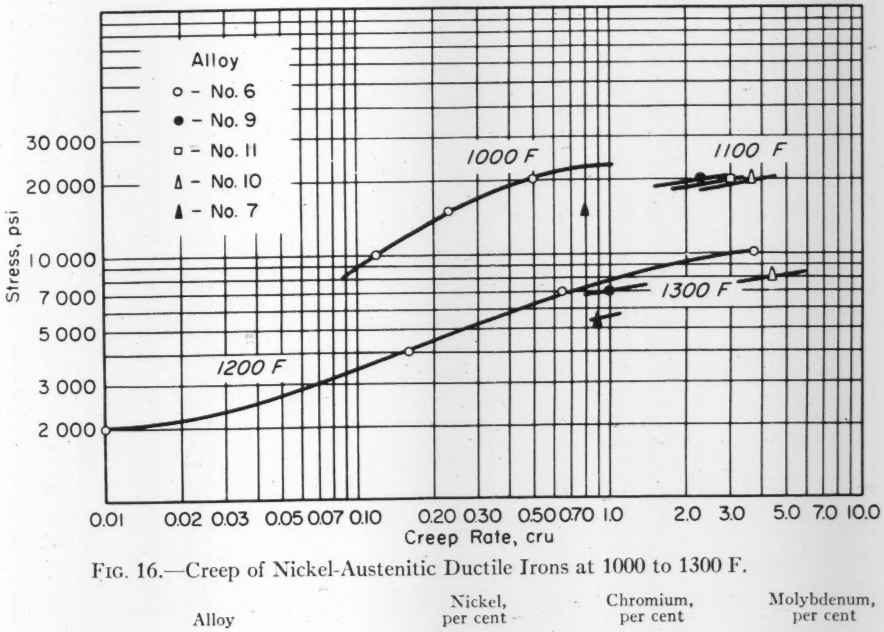

Fig. 16.—Creep of Nickel-Austenitic Ductile Irons at 1000 to 1300 F.

| Alloy | Nickel, per cent | Chromium, per cent | Molybdenum, per cent |
|---|---|---|---|
| No. 6 | 20.48 | 2.36 | .... |
| No. 9 | 30.18 | 3.05 | 0.95 |
| No. 11 | 35.21 | 2.61 | 0.73 |
| No. 10 | 35.17 | 2.85 | .... |
| No. 7 | 20.56 | 1.70 | 0.71 |

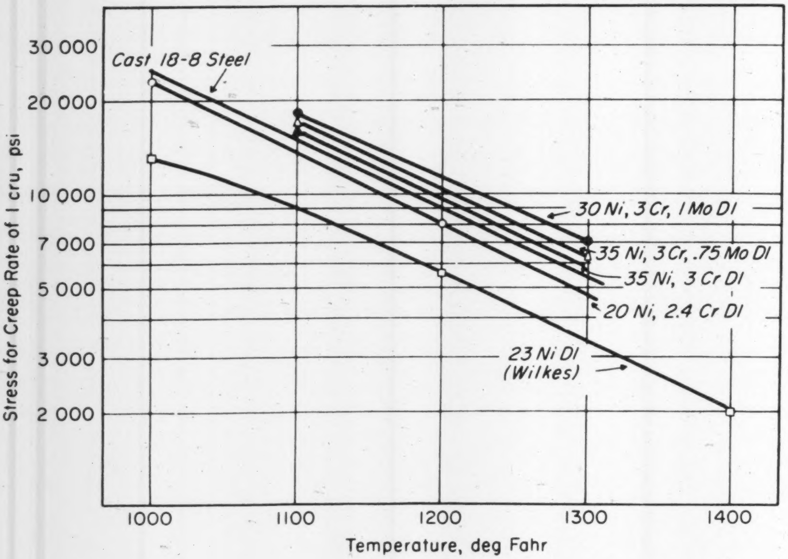

FIG. 17.—Comparison of Creep Strengths of Nickel-Austenitic Ductile Irons with Creep Strength of Cast 18-8 Stainless Steel.

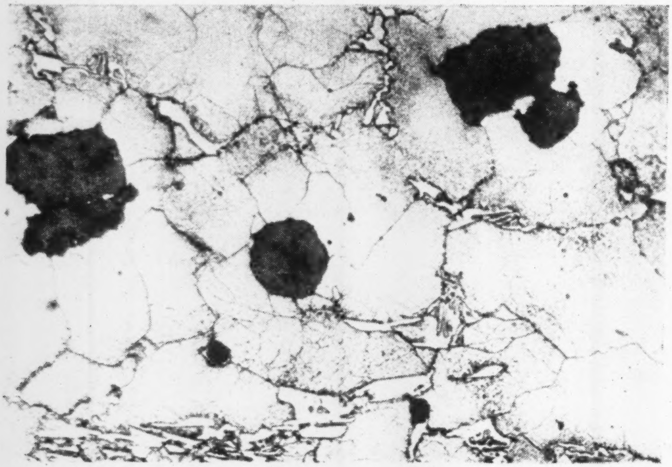

FIG. 18.—Microstructure of Austenitic Ductile Iron Alloy No. 6 after Creep Testing at 1000 F for 5080 hr. Nital Etched.

in alloy content causes a moderate increase in the creep strength of these austenitic irons. For example, the 23 per cent nickel iron of Wilks *et al* (**2**) showed a creep rate of 1 cru at 1200 F under a stress of 5700 psi, whereas the 30 per cent nickel, 3 per cent chromium, 1 per cent molybdenum heat tested in the present work carried 11,000 psi for the same rate.

The creep properties of this group of alloys are similar to those of cast 18-8 stainless steel (8) as is shown by Fig. 17. The irons containing 30 and 35 per cent nickel are somewhat more creep resistant than cast 18-8 stainless steel, while the iron containing 20 per cent nickel is not quite as strong.

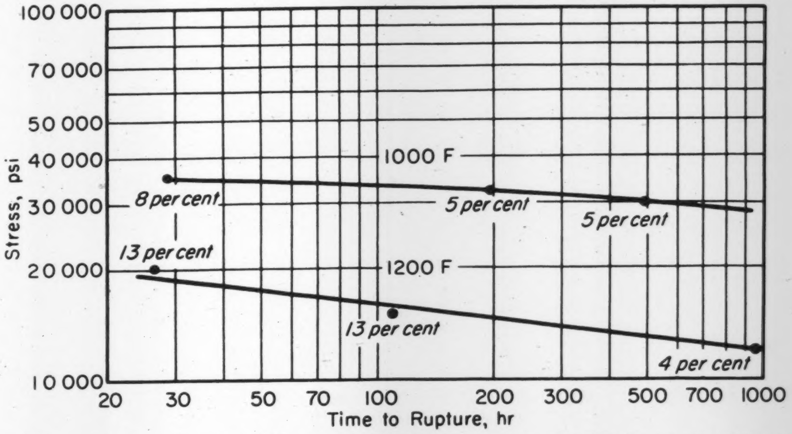

Fig. 19.—Rupture Data for 20 per cent Nickel, Austenitic Ductile Iron, Alloy No. 6.

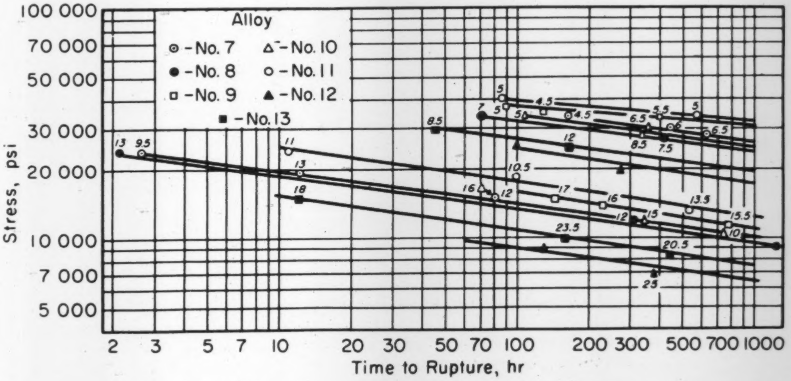

Fig. 20.—Rupture Data for Nickel-Austenitic Ductile Irons at 1100 F (upper group) and 1300 F (lower group).

| Alloy | Silicon, per cent | Nickel, per cent | Chromium, per cent | Molybdenum, per cent |
|---|---|---|---|---|
| No. 7 | 2.30 | 20.56 | 1.70 | 0.71 |
| No. 8 | 1.89 | 30.83 | 3.01 | .... |
| No. 9 | 1.99 | 30.18 | 3.05 | 0.95 |
| No. 10 | 1.51 | 35.17 | 2.85 | .... |
| No. 11 | 1.48 | 35.21 | 2.61 | 0.73 |
| No. 12 | 5.65 | 27.98 | 4.60 | .... |
| No. 13 | 5.66 | 27.37 | 4.36 | 0.87 |

During the long exposures at 1000 and 1200 F, extensive carbide precipitation occurred in the 20 per cent nickel samples (Fig. 18) which caused an increase in strength of the material. The following tension test results were obtained at room temperature on specimens of a 20 per cent nickel heat before

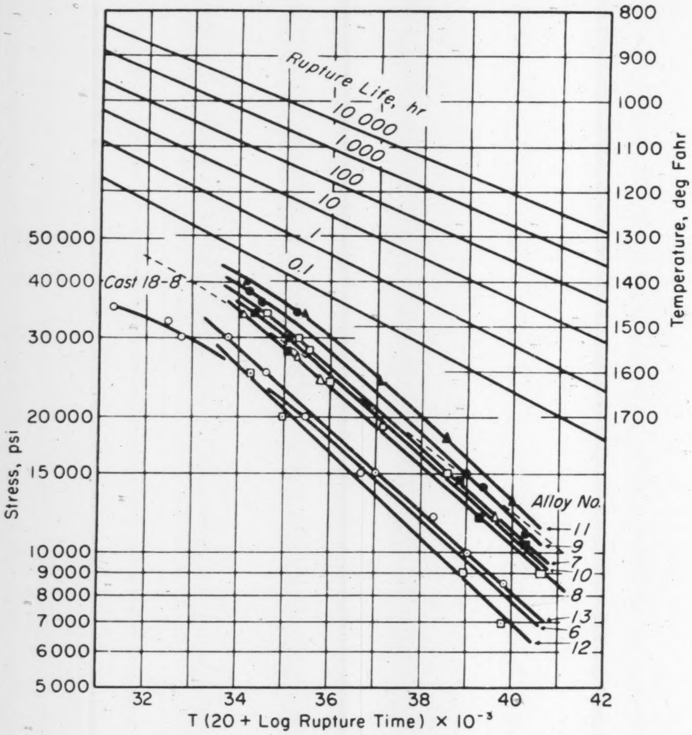

Fig. 21.—Comparison of Rupture Strengths of Nickel-Austenitic Ductile Irons with Data on Cast 18 per cent Chromium, 8 per cent Nickel Stainless Steel.

| Alloy | Nickel, per cent | Chromium, per cent | Molybdenum, per cent | Silicon, per cent |
|---|---|---|---|---|
| No. 11 | 35 | 2.6 | 0.75 | 1.5 |
| No. 9 | 30 | 3.0 | 1.0 | 2.0 |
| No. 7 | 20 | 1.7 | 0.7 | 2.0 |
| No. 10 | 35 | 3.0 | ... | 1.5 |
| No. 8 | 30 | 3.0 | ... | 2.0 |
| No 13 | 27 | 4.5 | 0.9 | 5.6 |
| No. 6 | 20 | 2.4 | ... | 2.0 |
| No 12 | 28 | 4.5 | ... | 5.6 |

and after a 5000-hr exposure at 1000 F under a stress of 10,000 psi.

| | Before | After |
|---|---|---|
| Ultimate strength, psi | 59 000 | 70 000 |
| Yield strength, psi | 35 000 | 45 500 |
| Elongation in 2 in., per cent | 11 | 4 |
| Rockwell hardness, B scale | 80 | 83 |

*Stress Rupture:*

Stress-rupture data for several nickel-austenitic ductile irons at 1000 to 1300 F are presented in Figs. 19 and 20. The stress to cause rupture of nickel-austenitic ductile iron after 1000 hr at 1100 F was found to vary from 18,500 psi[5] in an iron containing 20 per cent nickel and 2.4 per cent chromium to 24,500 psi for an iron containing 30 per cent nickel and 3 per cent chromium. Addition of 0.7 to 1 per cent molybdenum to these irons increased their respective 1000 hr rupture strengths by about 6000 psi. An increase in the nickel content of the iron from 30 to 35 per cent further increased the strength by 1500 to 2000 psi.

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[5] Interpolated value; see Fig. 21.

An increase in the silicon content of a 30 per cent nickel ductile iron from the usual 2 per cent level to 5.5 per cent with a simultaneous increase in chromium to 4.5 per cent was detrimental to the rupture strength, although such a change would greatly improve the corrosion resistance. The 1000 hr rupture stress at 1100 F for such an iron was 17,000 psi. The addition of 0.9 per cent molybdenum was not as effective in this alloy as in the others, for the rupture strength was increased by only 3000 psi.

At 1300 F the 1000-hr rupture stresses for this group of austenitic irons varied from 6500 psi for the 4.5 per cent chromium, 5.5 per cent silicon, 30 per cent nickel iron to 12,500 psi for the 35 per cent nickel type containing molybdenum. The relative performances of the various alloys remained approximately the same.

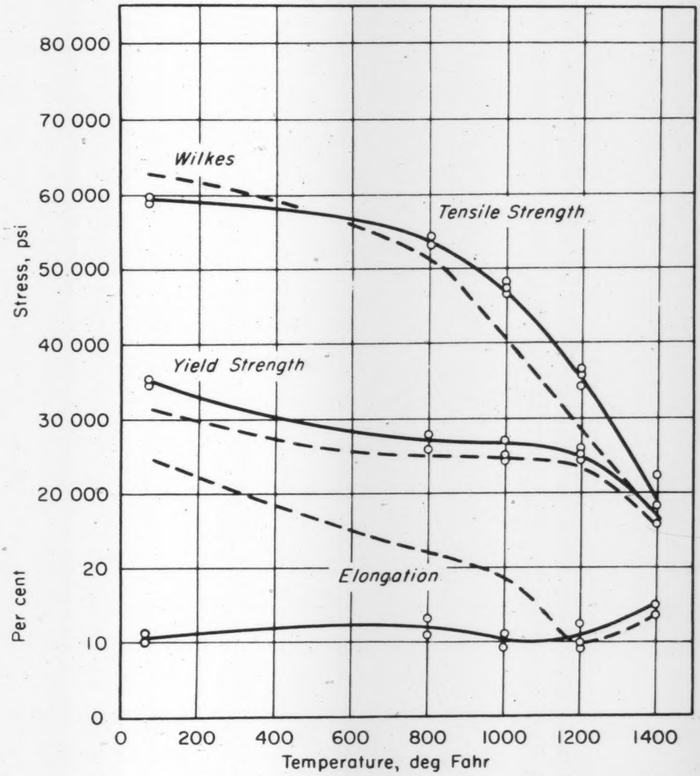

FIG. 22.—Tensile Properties of Nickel-Austenitic Ductile Iron at Elevated Temperatures.

All of the nickel-austenitic irons tested in this investigation contain larger amounts of alloying elements than the austenitic irons tested by Wilks *et al* (2) and on that account, have better rupture strengths. The range of rupture strengths exhibited by this group of alloys spans the reported values for cast 18 per cent chromium, 8 per cent nickel steel (9) at 1100 F. At 1300 F the strengths of 30 to 35 per cent nickel irons containing molybdenum are comparable to the strength of 18-8 stainless steel as is shown in Fig. 21. In this figure stress is plotted *versus* the parameter $T(20 + \log \text{rupture time}) \times 10^{-3}$, in which $T$ is in degrees Rankine and rupture time is in hours (10).

### *Short-Time Tensile Properties:*

Examples of the short-time tensile properties of this family of spheroidal graphite irons are shown in Fig. 22. The ultimate strength of the 20 per cent nickel, 2.4 per cent chromium melt decreased gradually from some 60,000 psi to 48,000 psi between room temperature and 800 F and then diminished to 20,000 psi at 1400 F. The elongation varied from 10 to 15 per cent over the same range. The chromium free iron investigated by Wilks *et al.* (2) possessed lower strength and higher ductility.

## CONCLUSIONS

1. Additions of molybdenum and copper to ductile iron greatly enhanced the creep resistance of the material. Increases in the amount of phosphorus present in the iron also were found to be beneficial. Creep properties similar to those of low-carbon steel were found for these irons.

2. An iron has been developed containing that has outstanding creep resistance in the annealed condition at 800 F.

| Total Carbon | Silicon | Manganese | Phosphorus | Nickel | Copper | Magnesium |
|---|---|---|---|---|---|---|
| 3.4 | 2.4 | 0.8 | 0.09 | 1 | 0.6 | 0.08 |

3. Heat-treated nickel-copper ductile irons were subject to mild precipitation hardening that caused strengthening during exposure at around 800 F.

4. The pearlite matrix structure was stable at 800 F during the longest test of some 6000 hr. At 1000 F, pearlite decomposed to ferrite and graphite.

5. The creep resistance of austenitic ductile irons containing 20 to 35 per cent nickel and 2 to 3 per cent chromium was greatly superior to that of the ferritic types. The addition of molybdenum to these irons was also highly beneficial. The creep properties of the group of irons were similar to those reported for cast 18 per cent chromium, 8 per cent nickel steel.

6. Long-time heating of austenitic irons at 1000 to 1200 F causes a precipitation of finely dispersed carbide that results in mild strengthening.

### *Acknowledgment:*

The authors acknowledge the assistance of many associates at the International Nickel Co. Research Laboratory, particularly A. M. Talbot and E. P. Sadowski under whose supervision the creep and stress-rupture tests were run. The authors are grateful to the International Nickel Co., Inc., for permission to publish the results of these tests.

## REFERENCES

(1) M. S. Saunders and M. J. Sinnott, "Some Properties of Nodular Iron at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 45, p. 362 (1953).

(2) C. R. Wilks, N. A. Matthews, and R. W. Kraft, Jr., "Elevated Temperature Properties of Ductile Cast Irons," *Transactions*, Am. Soc. Metals, Vol. 47, p. 611 (1955).

(3) F. G. Sefing, "Nickel Austenitic Ductile Irons," *Transactions*, Am. Foundrymen's Soc., Vol. 63, p. 638 (1955).

(4) F. B. Foley, "Mechanical Properties at Elevated Temperatures of Ductile Cast Iron," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 78, No. 7, Oct., 1956, p. 1435.

(5) G. R. Brophy and D. E. Furman, "The Cyclic Temperature Acceleration of Strain in Heat Resisting Alloys," *Transactions*, Am. Soc. Metals, Vol. 30, p. 1115 (1942).

(6) "Report on the Strength of Wrought Steels at Elevated Temperatures," Am. Soc. Testing Mats. (1950) (Issued as separate publication *ASTM STP No. 100*).

(7) Steel Castings Handbook, Steel Founders' Soc. America, p. 154 (1941).

(8) Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys, Am. Soc. Testing Mats., (1938). (Issued as separate publication *ASTM STP No. 37*).

(9) "Report on Elevated-Temperature Properties of Stainless Steels," Am. Soc. Testing Mats. (1951). (Issued as separate publication *ASTM STP No. 124*).

(10) F. R. Larson and J. Miller, "A Time-Temperature Relationship for Rupture and Creep Stresses," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 19, p. 765 (1952).

## DISCUSSION

MR. SAMUEL J. ROSENBERG.[1]—Some excellent information on the properties of ductile cast iron has been given, but I wish to comment on the method of plotting creep rate.

I believe it is wrong to plot creep rate in terms of 1 per cent per 10,000 hr unless the test has actually been carried to 10,000 hr. Even though it is a rate of which we are speaking, somehow the impression is conveyed that the test was conducted for that length of time. We may have equivalent creep rates of 0.01 per cent in 100 hr, or 0.1 per cent in 1000 hr, but that is no sign that we will have 1 per cent in 10,000 hr. By that time the specimen may have elongated several per cent and broken. Perhaps we should adopt some method of indicating the total time that the specimen was in the second stage of creep.

MR. R. D. SCHELLENG (*author*).—I am in complete agreement with your comments. In the paper the data are presented in terms of a creep rate unit (abbreviated cru) which we define as 0.0001 per cent per hr. In this presentation I used the 1 per cent in 10,000 hr unit which, perhaps erroneously, I believed was a more universally used and hence more readily understood term for expressing creep rates, although less rigorous than the per cent per hr term.

MR. ROBERT MATTERS.[2]—The authors' publication of this information on nodular irons is appreciated. It is hoped that the work will continue since the paper is more a general survey than definitive with regard to the high-temperature properties of nodular irons. For example, the power industry is interested in lower creep rates than the 0.0001 per cent per hr generally reported in the paper.

The authors have expressed surprise that the creep rates of ferritic irons containing nodular graphite and containing plate graphite are similar for similar compositions. This similarity of creep rate should be expected since the creep rate would be determined largely by the properties of the matrix. The rupture properties of nodular irons—ductility in rupture and rupture strength—should be higher than the rupture properties of flake graphitic irons. Probably the greatest difference in rupture properties will be observed at the higher temperatures.

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[1] Metallurgist, National Bureau of Standards, Washington, D. C.

[2] Assistant Director of Research, Allis-Chalmers Manufacturing Co., Milwaukee, Wis.

Work done in this laboratory on the properties of unalloyed nodular irons, with only trace amounts of nickel or copper, containing approximately 0.1 per cent phosphorus indicated that embrittlement occurred during the creep test. Have the authors observed any indications of embrittlement of their nodular irons during the creep tests?

MR. SCHELLENG.—Our investigations of the ductility of the material after testing were somewhat limited and we did not find the embrittlement which you describe.

MR. MATTERS.—It is not suggested that all nodular irons would be embrittled by exposure to creep, at elevated temperatures. Review of the laboratory data indicates that one high-phosphorus heat No. 846 was severely embrittled (23 per cent elongation before creep test, 2 per cent elongation after creep test), whereas the second high-phosphorus heat, heat No. 1270, was practically unaffected, with $20\frac{1}{2}$ per cent elongation before test, $21\frac{1}{2}$ per cent elongation after test. It is probable that this variation in behavior was caused by an interaction between phosphorus content and melting stock. The low-phosphorus heat No. 879 produced from the same melting stock as heat No. 846 lost some ductility during creep tests (28 per cent elongation before test, 17 per cent elongation after test). The low-phosphorus heat No. 1282 produced from the same melting stock as heat No. 1270 was also substantially unaffected by creep testing (22 per cent elongation before test, 22.5 per cent elongation after test). The tests performed in this laboratory confirmed the authors observation that the creep strength of the nodular irons was increased by an increase in phosphorus content. At a stress of 25,000 psi at 700 F, the minimum creep rate for the high-phosphorus heat No. 1270 was 0.039 per cent per 1000 hr and for the low-phosphorus heat 1282 was 0.75 per cent per 1000 hr.

# THERMAL SHOCK RESISTANCE OF HIGH TEMPERATURE ALLOYS*

By F. L. Muscatell,[1] E. E. Reynolds,[1] W. W. Dyrkacz,[1] and J. H. Dalheim[1]

## Synopsis

A new thermal shock or thermal fatigue testing apparatus and procedure simulating gas turbine service conditions was developed and is herein described. Results are presented for a number of heat-resistant superalloys and stainless steels showing the effects of test conditions and material variables. In general, wrought alloys were more resistant to thermal shock than cast alloys, and cobalt-base alloys were more resistant than either nickel or iron base alloys.

The evaluation of the influence of rapid repeated temperature changes on metals has been a challenging problem to material and design engineers connected with modern power plants. One such power plant is the aircraft gas turbine in which failure of a part by any cause could be catastrophic. In general, engineering background is sufficient with respect to mechanical and physical properties to the extent that safe, practical applications of materials can be assured. However, evaluation of such obscure effects as sudden alternating stresses from thermal gradients leave some doubt as to the margin of design safety.

Work in the field of thermal shock resistance or thermal fatigue of alloys has been limited. Most of it has been carried out under arbitrary conditions of heating and cooling with the design of the specimen selected so as to give reasonable failure times.

Chenea *et al* (**1**)[2] have reported on the design of a test specimen which would simulate conditions in service. These authors subjected specimens, varying in shape, to repeated cycles of gradual heating and rapid cooling. Heating was accomplished by passing an electric current through the specimen and cooling by a forced air quench. They found a triangular test specimen with flattened edges most suitable. Thielsch (**2**) reported on the metallurgical factors promoting thermal fatigue or shock failures. He also gave results from preliminary tests on six cobalt-bearing high-temperature alloys. All six alloys had similar physical properties such as coefficient of linear expansion, thermal conductivity, and specific heat but they differed widely

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Senior Research Metallurgist, Associate Director of Research, Manager, Quality Control, and Chief Test Engineer, respectively, Allegheny Ludlum Steel Corp., Watervliet, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 962.

in thermal shock lives. Lardge (3) reported thermal shock data on a few nickel-base high-temperature sheet alloys. His results showed that the thermal shock life of Nimonic 75, 80A, 90, and Inconel is increased by decreasing the material thickness. Sipple (4) also reported thermal shock data on high-temperature sheet alloys under stress. These authors showed the essential variables encountered in running thermal shock tests.

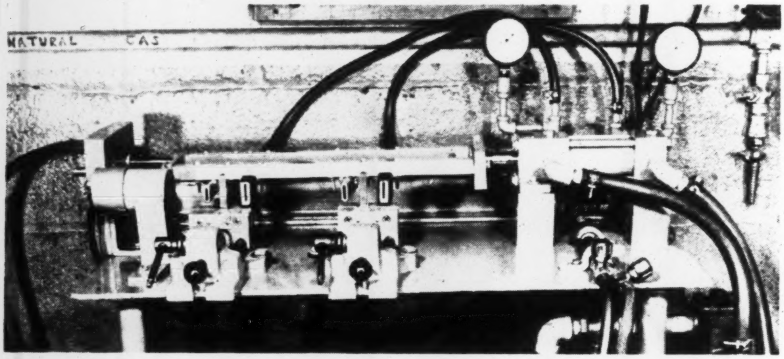


FIG. 1.—Thermal Shock Testing Unit.

The present work was initiated as a result of the need for further test methods to simulate service conditions and to evaluate various superalloys and stainless steels. This need was specifically outlined by the Gas Turbine Panel of the Joint ASTM-ASME Committee on the Effect of Temperature on the Properties of Metals. The task was assigned to Sub-Panel GTP-5 consisting of W. W. Dyrkacz, chairman, B. O. Buckland, A. W. F. Green, B. J. Lazan, S. S. Manson and E. L. Olcott. A new apparatus at The Research Laboratory of Allegheny Ludlum Steel Corp. was available for this project. This apparatus and the test method had previously been devised after a review of past work and established service needs. Members of the panel and others contributed test materials of current and future commercial interest.

## APPARATUS AND PROCEDURE

The thermal shock test involved repeated heating of a limited portion of the edge of a triangular shaped specimen in a propane-air flame from a Selas burner to a certain maximum temperature and then suddenly cooling this edge in a blast of compressed air. One view of the thermal shock unit is shown in Fig. 1. It consists of a steel framework to which is attached a control box that houses a cycle counter, relays, and switches necessary for automatic operation. Solenoid air valves and pneumatic pistons are used to move the burner and cooling nozzle back and forth in front of the test specimen and to turn the air blast off and on. The temperature time cycle of the test is controlled automatic-

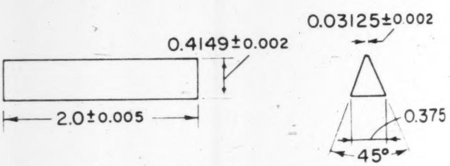


FIG. 2.—Thermal Shock Test Specimen.

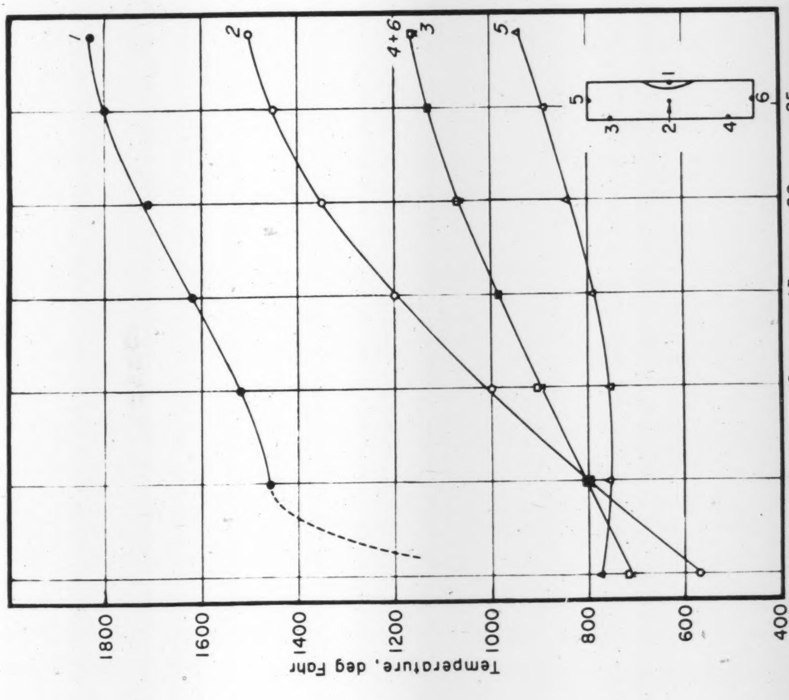


FIG. 3.—Thermal Gradient of Thermal Shock Test Specimen During Heating Time.

Heating cycle—29 sec heat, 6.5 sec cooling.

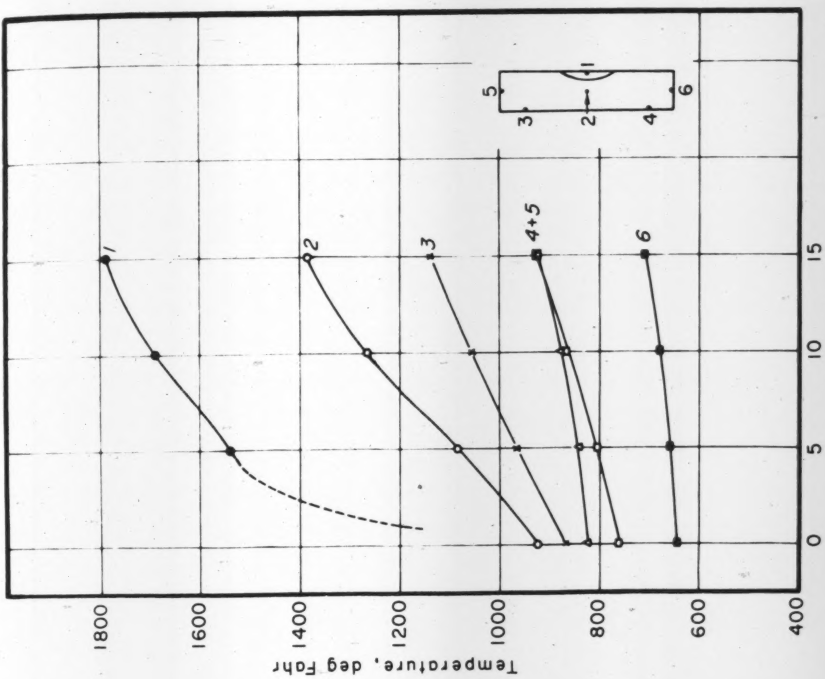


FIG. 4.—Thermal Gradient of Thermal Shock Test Specimen During Heating Time.

Heating cycle—15 sec heat, 3 sec cooling.

ally by the use of a Minneapolis Honeywell radiamatic head focused on the test edge. The radiamatic head acts through a high-speed Minneapolis Honeywell Circular Chart Electronik Potentiometer. The burner and controller are adjusted to obtain the desired edge temperature, which is measured with an optical pyrometer. The unit tests two specimens at a time. Specimen shape, test procedures, and conditions were selected to give failure in a reasonable time period. Each specimen is checked for external defects with a dye penetrant before testing.

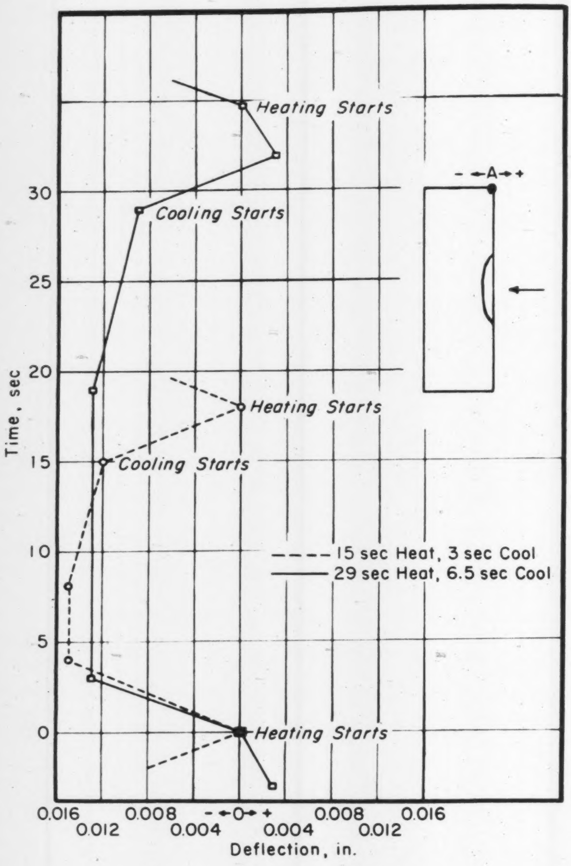


FIG. 5.—Deflection of a Test Specimen at Point *A* During a Thermal Cycle.

The triangular test specimen is shown in Fig. 2. Bar stock is machined and ground into an isosceles triangle cross section with an apex angle of 45 deg. The leading edge is then ground longitudinally to a flat surface $\frac{1}{32}$-in. wide. A Norton wheel (38 A 60 – H 8 VBE) was employed during the grinding operations resulting in a highly polished surface measuring less than 15 microinches (root mean square) on a surface indicator. This sample was decided upon after extensive preliminary experiments with angles and contours.

Thermal shock failures were determined by inspecting the specimen under a 20 × binocular microscope every 500 cycles until cracking began and every 50 cycles after the appearance of the initial crack. Failure was considered reached when a crack had proceeded completely across the $\frac{1}{32}$-in. wide test edge. All efforts to record the failure in the specimen automatically have been unsuccessful.

The thermal cycle can be varied from room temperature to any temperature up to 2000 F. A typical cycle is from room temperature to 1800 F, which requires approximately 20 sec, 17 for heating and 3 for cooling. Additional tests have been run with the maximum temperatures of 1600 and 1900 F. Tests run from room temperature to 1600 F require approximately 10 sec for heating and 3 sec for cooling. The 1600 F tests were run with a lower heating rate but approximately the same cooling rate as given above for the 1800 F test.

Both temperatures and deflections were measured during two different thermal cycles. Temperatures of the test specimen were measured at the various locations shown in the diagrams in Figs. 3 and 4. The temperature at the test edge was read by an optical pyrometer. All others were read by a thermocouple.

The thermal-time cycles during heating, for two cycles of 15 and 29 sec heating periods, are given in Fig. 5. Cooling periods were 6.5 and 3 sec, respectively, for the two cycles.

The deflection of the top of the test edge of the specimen during heating and cooling is also shown in Fig. 5. A very marked deflection, serving to make the test edge convex, occurred very shortly after the start of heating. This deflection remained the same for some time followed by a gradual decrease which was then relieved rapidly during cooling.

The front test edge of the specimen immediately elongated 0.002 in. when the heat was applied and contracted again on cooling. There was no observable bulging of the test edge during the heating cycle.

## TEST MATERIALS

The nominal chemical compositions of the alloys tested are given in Table I. Both cast and wrought alloys were included in the program and several alloys were tested in both forms. Heat treatments used on the wrought alloys are given in Table II.

## TEST RESULTS AND DISCUSSION

Early tests showed that test specimen failures produced on this thermal shock testing apparatus were similar to those occurring in service, and, moreover, service parts tested in the apparatus failed in a similar way to the test specimens. The microstructures in Fig. 6 show a cracked test specimen in comparison with a bucket that failed in service. The test specimen had approximately the same grain size as the turbine bucket before testing.

Buckland and Gatzemeyer (5) calculated the life of a gas turbine bucket in service by utilizing the results of these thermal fatigue tests and L. Coffin's (6) equation:

$$N = \left(\frac{K}{E_p}\right)^n$$

where:

$K$ = a constant varying for each material and depending on the ductility,

$N$ = number of cycles to failure,

$E_p$ = maximum localized plastic strain, and

$n$ = exponent assigned a value of 2 from previous tests.

TABLE I.—NOMINAL COMPOSITION OF ALLOYS IN PER CENT.

A. CAST ALLOYS

| Type | Carbon | Manganese | Silicon | Chromium | Nickel | Cobalt | Molybdenum | Tungsten | Titanium | Columbium + Tantalum | Aluminum | Iron | Boron | Other | Supplier |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| S-816 | 0.40 | 1.2 | 0.5 | 20 | 20 | Balance | 4 | 4 | .. | 4 | .. | 2 |  | .. | Allegheny Ludlum Steel Corp. |
| X-40 (HS-31) | 0.40 | 0.6 | 0.6 | 25 | 10 | Balance | .. | 8 | .. | .. | .. | 1 |  | .. | Haynes Stellite Co. |
| Vitallium (HS-21) | 0.40 | 0.6 | 0.6 | 26 | 3 | Balance | 5 | 4 | .. | 4 | .. | 1 |  | .. | Haynes Stellite Co. |
| HE-1049 | 0.40 | 0.7 | 0.3 | 25 | 10 | Balance | .. | 15 | .. | .. | .. | 0.7 |  | .. | Allegheny Ludlum Steel Corp. |
| GMR-235 | 0.10 | 0.5 | 0.5 | 15 | Balance | .. | 5 | .. | 2.3 | .. | 2 | 10 | 0.2 | .. | Haynes Stellite Co. |
| Guy | 0.14 | 0.4 | 0.8 | 15 | Balance | .. | 5 | .. | .. | 2 | 5 | 5 | 0.5 | .. | Allegheny Ludlum Steel Corp. |
| Refractalloy 80 | 0.40 | 0.7 | 1.0 | 20 | 20 | 30 | 10 | .. | 4 | .. | .. | .. | .. | .. | Bureau of Ships, Dept. of the Navy, Washington, D. C. |
| 304 | 0.014 | 0.96 | 0.24 | 20.64 | 10.92 | .. | 0.04 | .. | .. | .. | .. | Balance |  | Copper, 0.13 | Haynes Stellite Co. |
| 310 | 0.10 | 0.72 | 0.87 | 23.61 | 20.05 | .. | 0.09 | .. | .. | .. | .. | Balance |  | Copper, 0.08 | Haynes Stellite Co. |
| 316 | 0.11 | 1.15 | 0.60 | 16.88 | 12.98 | .. | 1.68 | .. | .. | .. | .. | Balance |  | Copper, 0.08 | Haynes Stellite Co. |
| 347 | 0.08 | 1.26 | 0.64 | 19.45 | 11.38 | .. | 0.04 | .. | .. | .. | .. | Balance |  | Copper, 0.11 | Haynes Stellite Co. |
| R-27 | 0.13 | 1.09 | 0.41 | 9.98 | 9.22 | Balance | 4.93 | 10.13 | .. | 0.74 | .. | 1.17 |  | .. | Allegheny Ludlum Steel Corp. |

B. WROUGHT ALLOYS

| Type | Carbon | Manganese | Silicon | Chromium | Nickel | Cobalt | Molybdenum | Tungsten | Titanium | Columbium + Tantalum | Aluminum | Iron | Supplier |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| S-816 | 0.40 | 1.5 | 0.5 | 20 | 20 | Balance | 4 | 4 | .. | 4 | .. | 3 | Allegheny Ludlum Steel Corp. |
| S-590 | 0.40 | 1.5 | 0.5 | 20 | 20 | 20 | 4 | 4 | .. | 4 | .. | Balance | Allegheny Ludlum Steel Corp. |
| L-605 (HS-25) | 0.12 | 1.5 | 1.0 | 20 | 10 | 51 | .. | 15 | .. | .. | .. | 1 | Allegheny Ludlum Steel Corp. |
| V-36 | 0.30 | 1.0 | 0.4 | 25 | 20 | Balance | 4 | 2 | .. | 2 | .. | 2 | Allegheny Ludlum Steel Corp. |
| Waspaloy | 0.08 | 0.7 | 0.5 | 20 | Balance | 14 | 4.5 | .. | 2.5 | .. | 1.3 | 1 | Allegheny Ludlum Steel Corp. |
| Hastelloy X | 0.10 | 0.5 | 0.6 | 20 | Balance | 1.0 | 9.5 | 1 | .. | .. | .. | 18 | Curtiss-Wright Corp. |
| N-155 (Multimet) | 0.10 | 1.5 | 0.88 | 20 | 20 | 20 | 3 | 3 | .. | .. | 1 | 4 | Curtiss-Wright Corp. |
| M-252 | 0.15 | 1.0 | 0.7 | 19 | Balance | 10 | 10 | .. | 2.5 | .. | 0.9 | 4 | Allegheny Ludlum and General Electric Co. |
| Nimonic 90 | 0.04 | 0.75 | 0.4 | 20 | 51 | 20 | 2 | .. | 2.5 | .. | 1.3 | .. | Allegheny Ludlum Steel Corp. |
| Udimet 500 | 0.10 | 1.0 | 0.75 | 19.5 | 54 | 13.5 | 4.3 | .. | 3.0 | .. | 3.0 | .. | General Electric Co. |
| G-157 | 0.10X | 1.5X | .. | 27 | Balance | .. | 1.5 | .. | 2 | .. | 0.75 | 6.0X | Allegheny Ludlum Steel Corp. |
| Jetalloy 209 | 0.03X | 0.50 | 0.50 | 20.0 | 10.0 | 50.0 | .. | 15.0 | 2.0 | .. | .. | 2X | Quebec Metallurgical Ind., Ltd. |
| Jet 1570 | 0.10 | 0.20X | 0.30X | 20 | Balance | 31 | .. | 6.5 | 4.1 | .. | .. | 2.5X | Universal Cyclops Steel Co. |
| 304 | 0.06 | 1.5 | 0.6 | 19 | 9 | .. | .. | .. | .. | .. | .. | Balance | Allegheny Ludlum Steel Corp. |
| 309 | 0.13 | 1.5 | 0.6 | 23 | 13 | .. | .. | .. | .. | .. | .. | Balance | Allegheny Ludlum Steel Corp. |
| 310 | 0.06 | 1.5 | 0.6 | 25 | 20 | .. | .. | .. | .. | .. | .. | Balance | Allegheny Ludlum Steel Corp. |
| 316 | 0.07X | 1.5 | 0.6 | 17 | 13 | .. | 2.5 | .. | .. | .. | .. | Balance | Allegheny Ludlum Steel Corp. |
| 347 | 0.05 | 1.5 | 0.6 | 18 | 10 | .. | .. | .. | .. | .. | .. | Balance | Allegheny Ludlum Steel Corp. |

They predicted a turbine blade life of approximately 4000 starting and stopping cycles in a locomotive engine. It was observed in actual service that approximately 2000 starts and stops caused failure of the buckets by thermal cracking. They felt this to be a good correlation since the additional cycles caused in operation on cutting back rapidly from full load to no load are unknown.

TABLE II.—HEAT TREATMENTS OF WROUGHT MATERIALS USED FOR THERMAL SHOCK TESTS.

| Alloy | Heat Treatment, Temperature, deg Fahr |
|---|---|
| S-816 | 2150 for 1 hr and WQ[a] + 1400 for 16 hr and AC[b] |
| S-590 | 2150 for 1 hr and WQ + 1400 for 16 hr and AC |
| L-605 (HS-25) | 2200 for 1 hr and WQ |
| V-36 | 2275 for 1 hr and WQ + 1400 for 16 hr and AC |
| Waspaloy | 1975 for 4 hr and AC + 1550 for 24 hr and AC + 1400 for 16 hr and AC |
| Hastelloy X | 2150 for ½ hr and WQ |
| N-155 (Mult.) | 2150 for 1 hr and WQ + 1500 for 4 hr and AC |
| M-252 | 1950 for 4 hr and AC + 1400 for 16 hr and AC |
| Nimonic 90 | 1985 for 8 hr and WQ + 1350 for 16 hr and AC |
| Udimet 500 | 2100 for 4 hr and AC + 1975 for 4 hr and AC + 1400 for 16 hr and AC |
| G-157 | 1975 for 1 hr and WQ + 1400 for 16 hr and AC |
| Jetalloy 209 | Forged, carburized, and solution-treated |
| Jet 1570 | 2150 for 4 hr and AC + 1650 for 24 hr and AC |
| 304 | 2000 for ½ hr and WQ |
| 309 | 2000 for ½ hr and WQ |
| 310 | 2000 for ½ hr and WQ |
| 316 | Unknown |
| 347 | 2000 for ½ hr and WQ |
| Hastelloy R | Mill Anneal |

[a] Water-quenched.
[b] Air-cooled.

*Effect of Alloy Composition and Condition:*

The thermal shock lives of various superalloys in the wrought and as-cast conditions are shown graphically in Figs. 7 and 8, respectively. These bar graphs indicate the range of cycles to failure of from two to five tests per alloy and also the average of these results.

Of the superalloys, the cobalt-base alloys appeared to be superior to the nickel-base alloys in both the cast and wrought conditions.

There was in some cases, a rather wide scatter of results for a given alloy. Even with this scatter, it was still possible to establish a varying degree of thermal shock life in the superalloys.

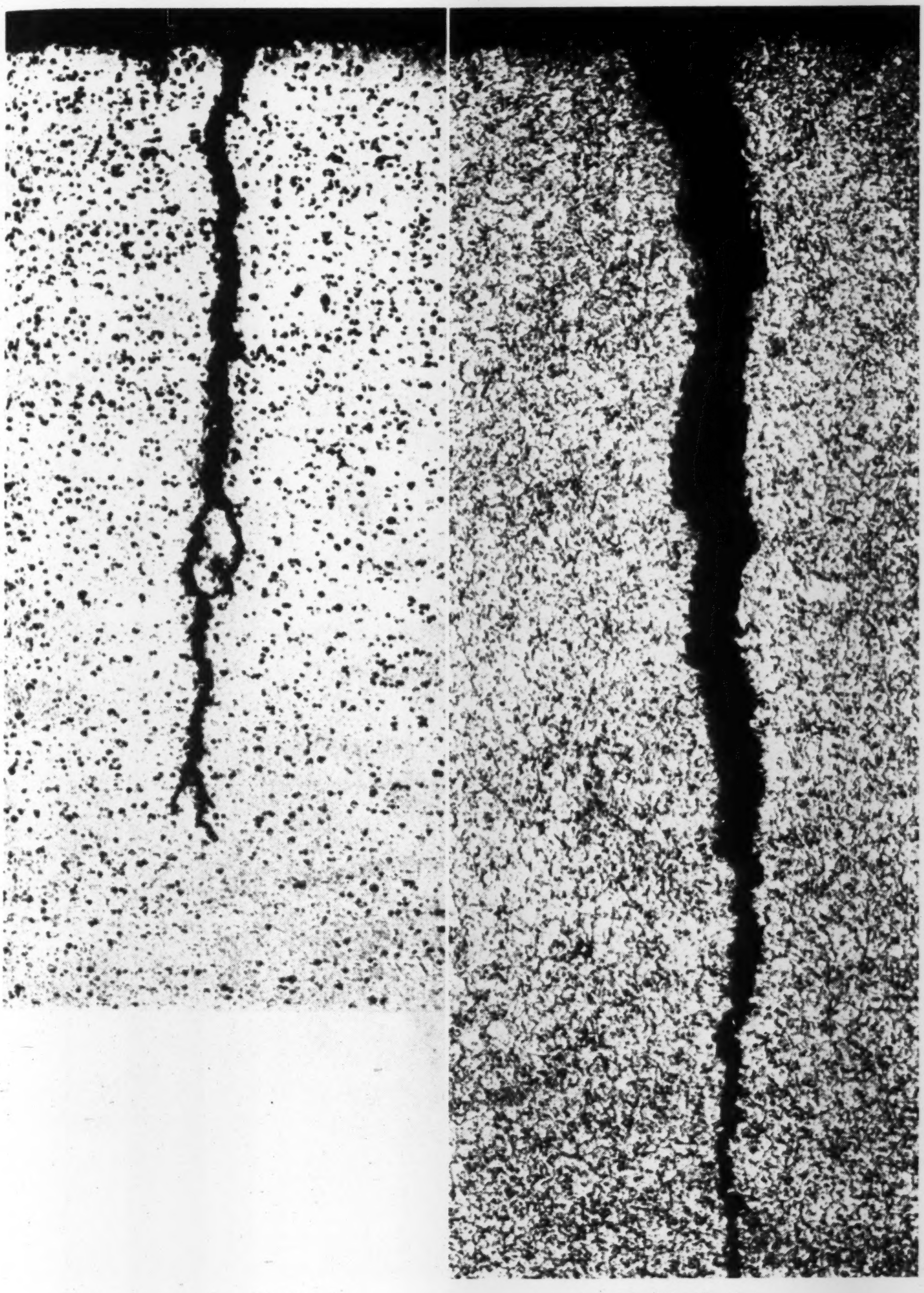
Attempts to correlate thermal shock properties with other properties of these materials such as thermal expansion, thermal conductivity, hot tensile properties, elastic modulus, impact properties, corrosion resistance, and structural conditions have not yielded any satisfactory relationship.

*Effect of Grain Size:*

The thermal shock lives of various wrought and cast stainless steels with varying grain size are shown in Fig. 9. It is noted that the larger the grain size, the lower the resistance to thermal cracking.

*Effect of Test Temperature:*

The effects of temperature on thermal shock life of several alloys are shown in Fig. 10. It is noted that the thermal shock is lowered, generally very markedly, with increasing maximum test temperature.



Wedge Sample from Bar Stock

Turbine Bucket

FIG. 6.—Thermal Cracks in S-590 Alloy (× 100).

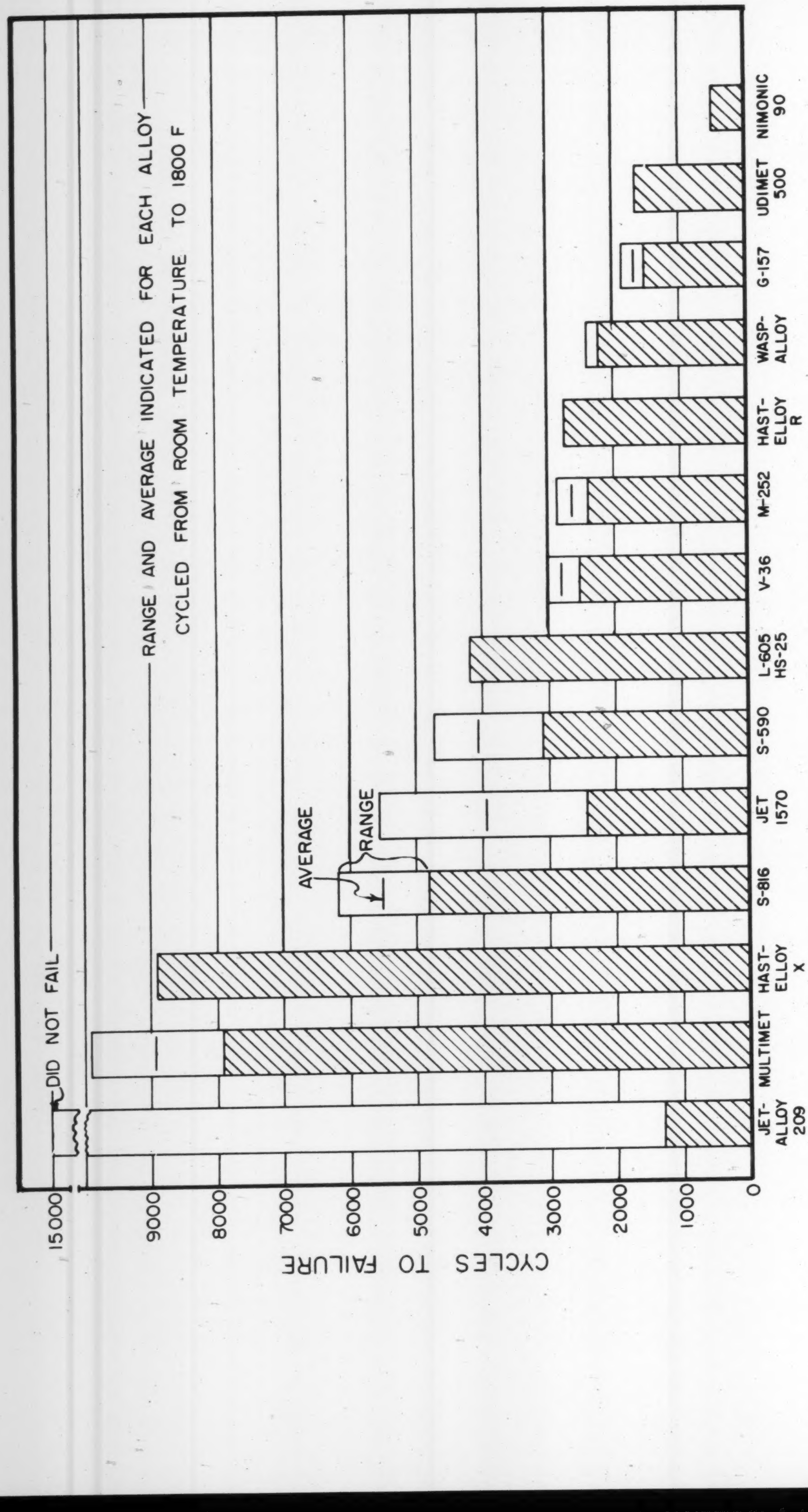


FIG. 7.—Cycles to Failure in Thermal Shock Testing of Wrought High-Temperature Alloys.

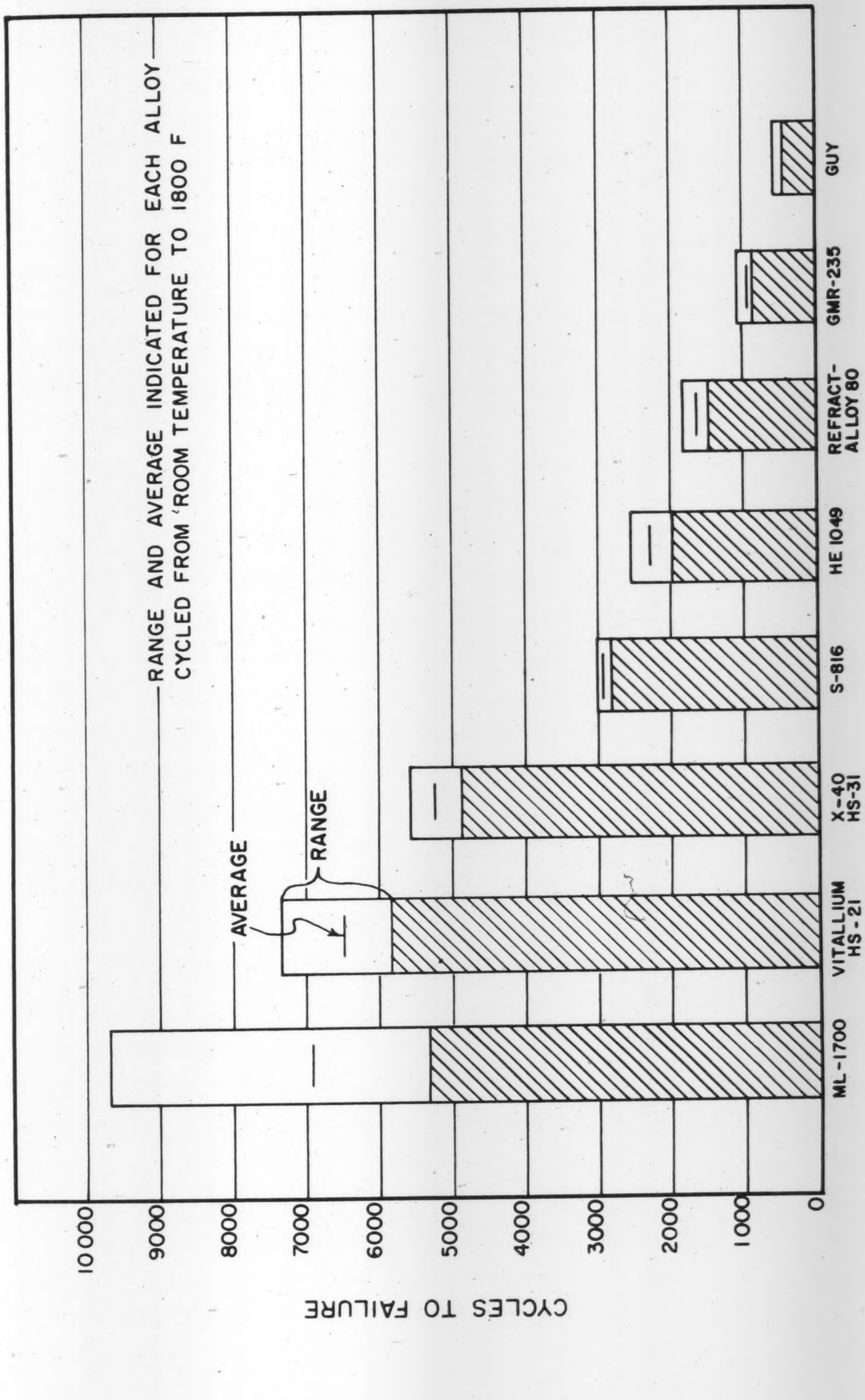


Fig. 8.—Cycles to Failure in Thermal Shock Testing of Cast High - Temperature Alloys.

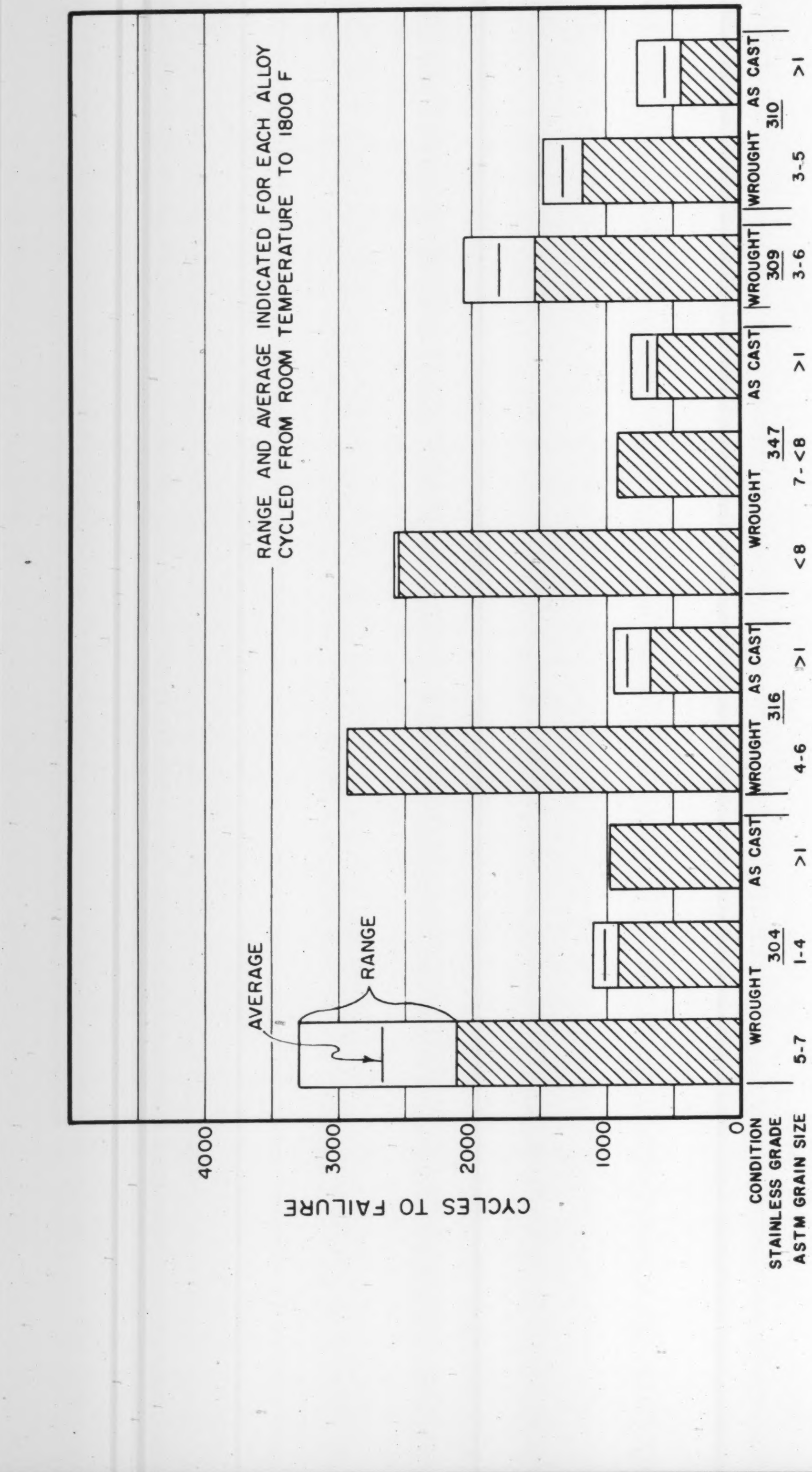


FIG. 9.—Effect of Grain Size and Condition on Thermal Shock Life of Stainless Steels.

An exception to this rule is afforded by one series of tests on Jetalloy 209 where failure occurred at 1600 F in 10,710 cycles whereas no failure occurred in 15,000 cycles at 1800 F maximum temperature. Jetalloy 209 is a low-carbon, cobalt-base superalloy which is forged and subsequently carburized and diffusion heat treated. It has been suggested that at the higher test temperature, decarburization of the material takes place more rapidly. This in turn apparently results in a greater decarburized zone with increased ductility. The decarburized region occurring on thermal shock testing at 1600 and 1800 F is shown in Fig. 11.

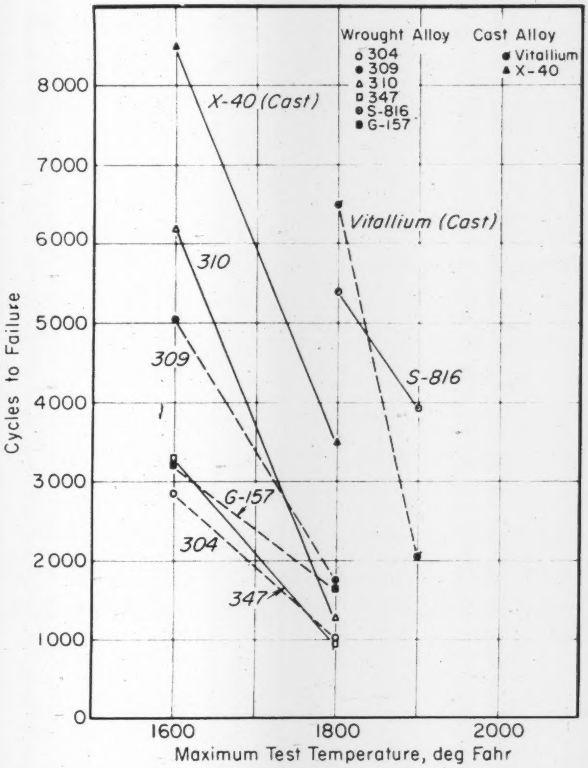
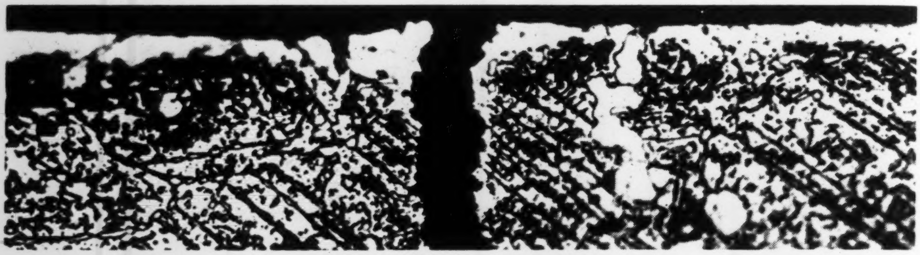


FIG. 10.—Effect of Maximum Temperature of Thermal Cycles on Thermal Shock Life. Minimum test temperature was room temperature.

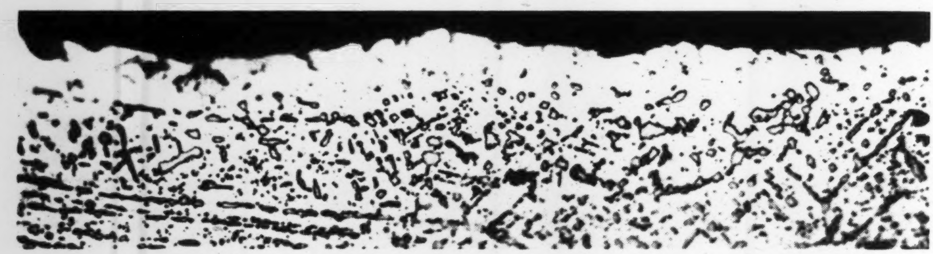
*Effect of Surface Conditions:*

*Effect of Surface Finish.*—Limited data on cast specimens of S-816, modified S-816, and R-27, as shown in Table III, indicate that the thermal shock life is affected adversely if the cast specimens are only partially ground or not ground at all. The ground specimens were surface ground followed by polishing on a wet grinding belt.

*Effect of Aluminum Coating.*—Test specimens of cast X-40 were coated with an aluminum protective coating as arranged by the Bureau of Ships. Two sets of specimens were coated by two processes. In process A, the aluminum was reportedly sprayed on the specimen and diffused for 2 hr at 1900 F. In process B, the specimen was hot dipped into the alloy used for coating and diffused for 20 min at 2060 F. The latter process formed a thin uniform alloy band overlaid by a smooth aluminum coating. The coating produced by process A was thicker but not as uniform. Microstructural examination revealed both coatings showed a



Maximum test temperature, 1600 F. Failure at 10,710 cycles.



Maximum test temperature, 1800 F. No failure at 15,000 cycles.

FIG. 11.—Jetalloy 209 Thermal Shock Specimens (× 500).

TABLE III.—EFFECT OF SURFACE FINISH ON THERMAL SHOCK LIFE OF CAST ALLOYS.

| Alloy | Heat | Cycles to Failure 80 to 1800 deg Fahr | | As Cast |
|---|---|---|---|---|
| | | Ground Surface | Test Edge Ground Only | |
| S-816 | F-456 | 2747<br>2991 | 2062<br>1837 | 1337<br>1337 |
| Average | | 2869 | 1949 | 1337 |
| S-816 0.10 per cent Carbon | F-457 | 2009<br>2009 | 1337<br>1480 | |
| Average | | 2009 | 1409 | |
| R-27 | F-496 | 2364<br>1909 | 1583<br>2028 | 1197<br>1397 |
| Average | | 2137 | 1805 | 1347 |

TABLE IV.—EFFECT OF COATINGS ON THE THERMAL SHOCK LIFE OF CAST X-40 (HS-31).

| Specimen | Heat B-1204 Temperature Cycle 80 to 1800 deg Fahr |
|---|---|
| | Cycles to Failure |
| As cast | 4 828<br>5 535 |
| Aluminum-coated by Process A | 4 840<br>4 840<br>5 025 |
| Aluminum-coated by Process B | 11 250[a]<br>6 240<br>9 543 |
| Chromium-coated | 3 596<br>2 205 |

[a] Did not fail.

varying degree of bonding along the specimen after testing. Results in Table IV show that process B did increase the thermal shock life whereas process A specimens did not show any effect.

*Effect of Chromium Coating.*—Additional test specimens of cast X-40 were chromallized through arrangements made by the Alco Products Co, Schenectady, N. Y. The chromallized operation was performed by the Chromalloy Corp., White Plains, N. Y. It is also noted in Table IV that the chromallized specimens had a lower thermal shock life than untreated specimens.

### *Effect of Melting Procedures:*

Several M-252 heats melted by different techniques including air, vacuum induction, vacuum and inert atmosphere consumable electrode melting were tested in order to determine the effect of melting variables upon the thermal shock properties of the alloy. The results given in Table V show no appreciable effect on thermal shock life by different melting techniques. All of the specimens tested had grain sizes in the range of ASTM Nos. 5 to 8.

### *Effect of Boron Additions to Cast Alloys:*

Increasing additions of boron decrease the thermal shock life of X-40, S-816 and Vitallium to a maximum test temperature of 1800 F as shown in Table VI.

TABLE V.—EFFECT OF MELTING ON THE THERMAL SHOCK LIFE OF M-252 ALLOY.

Heat Treatment: 1950 for 4 hr and air cooled + 1400 for 16 hr and air cooled
Temperature Extremes: 80 to 1800 F (cyclic)

| Heat | Type of Melt | Bar Size | Brinell Hardness | Cycles to Failure |
|---|---|---|---|---|
| 87-2-0051 | 10-ton arc furnace (Air Melt) | 1 3/16 in. round | 302/321 | 2756, 2326 |
| 4X-187 | Argon consumable-electrode | 3/4 in. round | 340 | 2818, 2356 |
| 4X-171 | Vacuum consumable-electrode | 3/4 in. round | 340 | 2831, 2582 |
| 3-267 | Vacuum induction | 7/8 in. round | 364 | 2500, 2100 |
| 874 | Vacuum induction | 3/4 in. round | 358 | 2300, 2132 |

TABLE VI.—EFFECT OF BORON ON THERMAL SHOCK LIFE.

| Alloy | Heat | Maximum Temperature Cycle, deg Fahr | Alloy Modification, per cent | Cycles to Failure |
|---|---|---|---|---|
| X-40 (HS-31) | F-691 | 1600 | 0.1 | 6050 |
| | F-594 | 1600 | 0.3 | 3900 |
| | F-593 | 1800 | | 3300 |
| | F-691 | 1800 | 0.1 | 2050 |
| | F-594 | 1800 | 0.3 | 1400 |
| S-816 | F-595 | 1600 | | 3750 |
| | F-596 | 1600 | 0.9 | 3140 |
| | F-595 | 1800 | | 2750 |
| | F-596 | 1800 | 0.9 | 1990 |
| Vitallium (HS-21) | F-373 | 1800 | | 6473 |
| | F-380 | 1800 | 0.2 | 3854 |

## Conclusions

1. The thermal shock resistance of the cobalt-base alloys was superior to that of nickel- and iron-base alloys.

2. Alloys were more resistant to thermal shock in the wrought than in the cast condition.

3. Thermal shock resistance of stainless steels increased with decreasing grain size.

4. An increase in the maximum test temperature generally lowered the thermal shock resistance of the alloy.

5. Surface condition had a definite influence on the thermal shock lives of certain cobalt-base superalloys.

6. There was no appreciable effect on the thermal shock life of M-252 melted

by different techniques, including air, vacuum induction, and vacuum and inert atmosphere consumable electrode melting.

7. The addition of boron to cast alloys X-40, S-816, and Vitallium lowered their thermal shock lives.

*Acknowledgments:*

The suggestions and stimulating discussion of members of Sub-Panel GTP-5 are gratefully acknowledged. For the helpful cooperation of all those who supplied materials as indicated in Table I, to G. R. Heckman and J. B. Gatzemeyer of the General Electric Co who supplied samples of shock failures from service, thanks are extended. The metallographic work of C. B. Craver and testing supervision by D. L. Wallace are also acknowledged.

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## REFERENCES

(1) P. F. Chenea, A. R. Bobrowsky, and L. L. Thomas, "Thermal Shock Investigation, Summary Report," University of Michigan, Wright Air Development Center, U. S. Air Force Contract AF 33(038)-21254, Project M949, Oct., 1952.

(2) H. Thielsch, "Thermal Fatigue and Thermal Shock," *Bulletin Series*, Welding Research Council, No. 10, April, 1952.

(3) H. E. Lardge, "Thermal Fatigue Testing of Sheet Metal," Symposium on Metallic Materials for Service at Temperatures above 1600 F, Am. Soc. Testing Mats., p. 146 (1955). (Issued as separate publication *ASTM STP No. 146.*)

(4) G. R. Sipple, "Progress Report on Thermal Shock Resistance of Various High Temperature Sheets Materials," *Report No. 811*, Allison Division, General Motors Corp. May, 1952.

(5) F. F. Buckland and J. B. Gatzemeyer, "Transient Temperature and Thermal Stress in Locomotive Gas Turbine Buckets," *ASME Paper No. 55-A-179*, Am. Soc. Mechanical Engrs., Nov., 1955.

(6) L. F. Coffin, Jr., "Study of the Effects of Cyclic Thermal Stresses on a Ductile Material," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 76, No. 6 (1954).

# DISCUSSION

Mr. D. O. Leeser.[1]—The authors are to be congratulated on the volume and quality of work they have done. They have contributed from their backlog of data a most valuable paper, which will not only assist the designer in a correct choice of materials by property data alone, but their significance of transient conditions which may occur during operation.

The authors made no reference to thermal shock produced by liquid metals, such as reported by other investigators.[2] Using liquid metals, thermal shock can be more severe even though the temperature range may not be quite as large as in gas systems. Assuming liquid metal operation between 1000 to 1100 F and a transient down to 500 F at a rate of between 200 and 400 F per sec, would you have any information, or would you care to speculate as to what might happen?

Mr. E. E. Reynolds (*author*).—Mr. Leeser's remarks are greatly appreciated. We have no experience with thermal shock resulting from exposure to liquid metals but we concur that the shock would tend to be more severe than with gases. We would speculate, however, that under the conditions stated, 1000 to 500 F at rates of 200 to 400 F per second, the shock conditions would not be too severe on metallic materials such as superalloys or stainless steels. Actually at these temperatures the stainless steels would be more appropriate than the superalloys.

Mr. A. W. F. Green.[3]—I would like to ask the author one question. He said that in the case of M-252 that there was no difference in thermal shock on M-252 material when it was air melted, vacuum melted, etc. We do not substantiate these data and have run many tests on material that was air melted, vacuum melted, consumable electrode melted. We find that in general there is an increase in thermal shock or thermal fatigue resistance in vacuum melted metal versus that produced by air-melting procedures.

Another matter for discussion concerned this question of grain size. We are now completing some data which indicate that in a very large grain, cast nickel-base alloy, we have reached thermal shock qualities at 2100 F not before obtained at much more than 1900 F. The material was vacuum melted in both cases except that the lower temperature graded material was normal fine grain as cast. It was interesting to hear the observations concerning boron because boron does give a grain boundary constituent, which under some conditions, especially if you go much over 0.10 per cent by compositional weight, forms an alloy which deteriorates in the grain boundaries at about 2125 to 2150 F.

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[1] Head, Materials Section, Atomic Power Development Associates, Detroit, Mich.

[2] R. A. Tidball and M. M. Schout, "Thermal Shocking Austenitic Stainless Steels with Molten Metal," *Transactions*, Am. Soc. Metals, Vol. 76, pp. 639–643 (1954).

[3] Technical Assistant to Executive Engineer, Allison Division of General Motors Corp., Indianapolis, Ind.

MR. REYNOLDS. We concur that there are many factors which affect life in these tests. We believe that fracture is essentially a fatigue failure. Grain boundaries, particularly those in which oxidation took place, would initiate fracture and then a typical fatigue type of failure would occur. Structure also had a significant effect on life.

When there were greater amounts of excess constituents, such as in a cast super alloy with considerable amount of carbon or boron, the more excess constituent the lower the thermal shock life.

We too expected an improvement in life with vacuum melting. Perhaps, however, we had a very clean air melted heat. We do know that in general the fatigue life of vacuum melted materials of the M-252 type is higher than that of air melts because of increased cleanliness.

# THERMAL SHOCK TESTING UNDER STRESS OF CERTAIN HIGH TEMPERATURE ALLOYS*

By F. K. Lampson,[1] T. C. Tsareff, Jr.,[2] and A. W. F. Green[2]

## Synopsis

Tests were made on eleven different high-temperature wrought alloys in sheet form at varying temperatures by first applying a known stress, then heating rapidly to a given temperature and cooling rapidly to temperatures approximating 100 to 120 F. Cycling was continued to failure, or if no failure occurred after a given number of reversals the test was stopped. All of the materials tested, that is, Tinidur, A-286, Discalloy 24, 19-9 DL, Timken 16-25-6, HS-88, N-155, S-590, V-36, WF-31, and L-605 showed excellent resistance to thermal shock at the temperatures used, namely, 1200, 1350, 1500, 1600, and 1800 F. However, as would be expected under the conditions of testing, the number of cycles to produce failure was a function of stress as calculated against short-time elevated temperature tensile strength, and the number of cycles to failure decreased as temperatures increased, especially in the range 1500 to, and including, 1800 F. Metallographic studies of all test specimens after testing showed that all fracturing initiated in and tended to follow grain broundaries—that is, were of the intergranular type—with evidence that preferential oxidation at the grain boundaries preceded actual failure. The work was done as an assignment from the Aviation Panel of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals. The test equipment was designed and built by the Allison Division of General Motors Corp.

The carrying out of the assignment to determine thermal shock resistance of certain high-temperature alloys in a wrought state and sheet form has provided information, which it is believed will add to the over-all accumulation of data and knowledge of the behavior of metals at high temperature while under stress.

The eleven high-temperature metals tested have been or are being used in the manufacture of turbojet engines and accessories thereto and similar power plants for aviation use. They are used also in other aircraft applications where resistance to elevated temperatures are involved, as well as in gas turbine development for other purposes.

The testing procedure used was developed with the knowledge that most of the parts and assemblies making up the various components of the modern turbojet aviation engine, especially in the "hot end," must serve not only the function of the part in question but actually become structural members in the assembly as a whole. They must, therefore, not only be able to show resistance to exposure to elevated temperatures but demonstrate ability to carry loads at such temperatures, often under high vibratory stress and, frequently, under severe reversals of both stress and temperature in relatively short periods of time. Since the very nature of turbojet engine functioning in aviation application involves large quantities of heated air, oxidation poses a vital problem. The testing procedure used took some cognizance of this.

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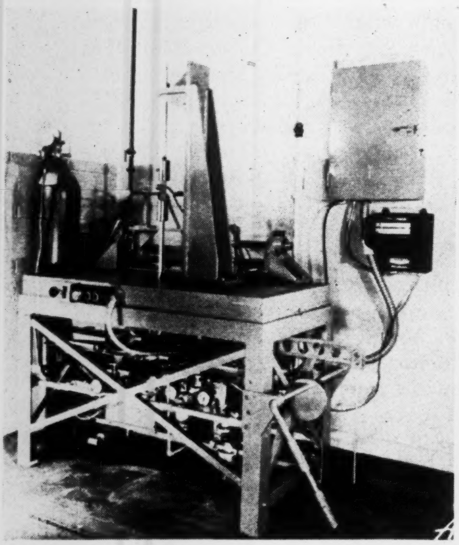


FIG. 1.—Thermal Shock Testing Machine.

If the tests produced no other result, it is believed that the hundreds of metallographic studies made during its conduct demonstrate that none of the materials designed for use at elevated temperatures is any better than its ability to resist oxidation, and that oxidation at elevated temperatures is again shown to be a function of grain-boundary attack. In other words, the inherent high-temperature strength of any material can only be properly utilized when the material either has adequate oxidation resistance or suitable surface protection is provided. Research and development of materials and processes for providing surface protection of metals should be continued.

## PROCEDURE

A machine was designed and constructed by Allison Division of General Motors Corp. to conduct thermal shock-resistance tests on various high-temperature alloys in sheet form in conjunction with a project originated by the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals. This machine is unique in that it imposes not only heating and cooling effects on a specimen but also maintains the specimen under a static axial stress during testing. A general view of the machine is shown in Fig. 1. The specimen is held in wedge-type grips and loaded by means of weights placed on a 10:1 lever arm receiver.

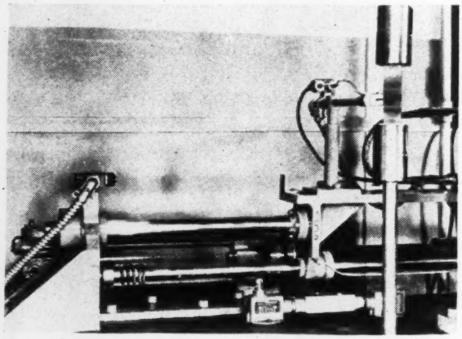


FIG. 2.—Position of Burner Nozzle in Relation to Specimen During Heating up Cycle.

The specimen is heated by combustion of a city gas compressed air oxygen mixture which is channeled through a ceramic nozzle and made to impinge on the specimen at an angle of approximately 70 deg, thus providing a heated area of a tapered type, approximately 1 in. wide spreading to approximately $1\frac{1}{4}$ in. across the width of the specimen.

The temperature is determined by means of a chromel-alumel thermocouple placed on the specimen at a point directly opposite the area of flame impingement, and indicated on a Brown recording potentiometer. The equipment is designed to provide automatic cycling such that testing is automatic after preliminary settings are established. A pneumatic system is used to actuate the movement from heating to cooling nozzles. Figure 2 shows the position of the burner nozzle in relation to the test specimen during a heating-up cycle. Figure 3 shows the cooling air nozzle impinging on the specimen after completion of the heating-up cycle. The temperature of the heated zone of the specimen is reduced rapidly to 100 to 110 F by such procedure. One or more cooling nozzles may be employed as desired. The maximum temperature used during conduct of the tests as detailed hereinafter was 1800 F.

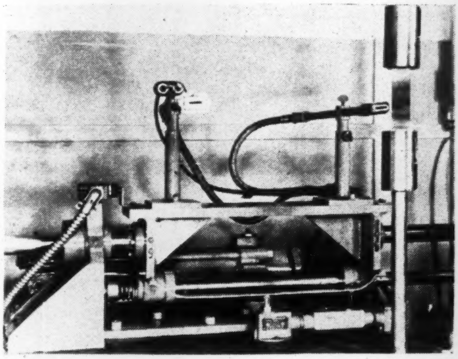


FIG. 3.—Position of Cooling Air Nozzle in Relation to Specimen after Heating Nozzle Has Been Removed.

A typical heating-and-cooling rate for the specimens tested as discussed in this paper is shown in Fig. 4. A precise heating-and-cooling rate relationship was established for each specimen size and material before starting an actual cycling test. It is obvious that no single heating-cooling rate could be used for all specimens. It can be seen that, with a test temperature of 1800 F, approximately 20 sec was required to cool the specimen to 110 F. Conversely, it took approximately 20 sec to heat the specimen to 1800 F.

*Details of Testing:*

The material for this investigation as supplied by the Aviation Panel of the Joint Committee, was nominally in the form of sheet stock 0.062 in. thick. The chemical analyses of the various materials are recorded in Table I. It can be seen that all of the alloys were iron-base except WF-31, V-36, and L-605, which were cobalt-base.

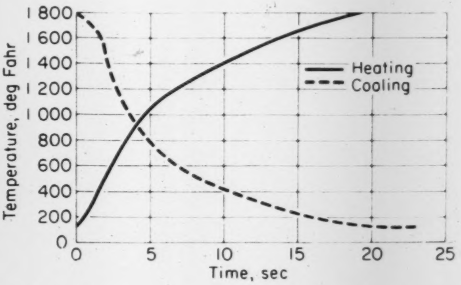


FIG. 4.—Typical Heating and Cooling Curves for the Sheet Metal Specimens.

The sheet stock was sheared into specimens 1 by 6 by 0.062 in. so that one set of the specimens was obtained with the long dimension parallel to the rolling direction and another set with the same dimension perpendicular to the sheet rolling direction. Specimens cut immediately adjacent to and following the same directional procedure were prepared from each sheet sample to obtain speci-

TABLE I.—CHEMICAL ANALYSES, PER CENT, OF HIGH-TEMPERATURE SHEET MATERIALS SUBMITTED BY JOINT ASTM-ASME COMMITTEE AVIATION PANEL FOR THERMAL SHOCK TESTING.

| Alloy | | Carbon | Manganese | Phosphorus | Sulfur | Silicon | Chromium | Nickel | Iron | Cobalt | Molybdenum | Tungsten | Titanium | Niobium | Aluminum | Others |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Tinidur | A[1] | 0.07 | 0.92 | 0.012 | 0.007 | 0.75 | 14.70 | 26.00 | Balance | ... | ... | ... | 1.90 | ... | 0.62 | ...... |
| | V[2] | 0.04 | 1.00 | 0.020 | 0.015 | 0.73 | 14.70 | 26.10 | Balance | ... | ... | ... | 2.26 | ... | 0.15 | ...... |
| A-286 | A | 0.03 | 1.63 | 0.017 | 0.017 | 0.98 | 15.4 | 26.70 | Balance | ... | 1.37 | ... | 1.60 | ... | 0.165 | 0.29 V |
| | V | 0.02 | 1.57 | 0.019 | 0.015 | 0.99 | 15.24 | 26.28 | Balance | ... | 1.25 | ... | 1.67 | ... | 0.170 | 0.32 V |
| Discalloy 24 | A | 0.05 | 1.41 | ... | ... | 0.85 | 13.70 | 25.60 | Balance | ... | 4.40 | ... | 2.30 | ... | 0.55 | ...... |
| | V | 0.036 | 1.38 | ... | ... | 1.00 | 13.50 | 26.20 | Balance | ... | 3.91 | ... | 1.61 | ... | 0.11 | ...... |
| 19-9 DL | A | 0.29 | 0.91 | 0.016 | 0.018 | 0.80 | 20.00 | 9.70 | Balance | ... | 1.50 | 1.63 | 0.30 | 0.48 | ... | |
| | V | 0.27 | 1.22 | 0.017 | 0.009 | 0.41 | 19.28 | 9.99 | Balance | ... | 1.35 | 1.43 | 0.30 | 0.50 | ... | 0.37 Cu |
| Timken 16-25-6 | A | 0.10 | 1.64 | ... | ... | 0.40 | 16.70 | 25.00 | Balance | ... | 6.65 | ... | ... | ... | ... | 0.13 $N_2$ |
| | V | 0.084 | 1.70 | 0.017 | 0.011 | 0.40 | 15.93 | 25.00 | Balance | ... | 6.00 | ... | ... | ... | ... | 0.132 $N_2$ |
| Haynes No. Stellite 88 | A | 0.06 | 1.24 | 0.011 | 0.015 | 0.62 | 12.80 | 15.70 | Balance | ... | 2.60 | 0.68 | 0.48 | ... | ... | |
| | V | 0.07 | 1.40 | 0.013 | 0.006 | 0.52 | 12.62 | 15.04 | Balance | ... | 2.42 | 0.84 | 0.50 | ... | ... | 0.14 B |
| N-155 | A | 0.07 | 1.15 | ... | ... | 0.54 | 20.90 | 20.20 | Balance | 20.60 | 4.00 | 2.78 | ... | 0.88 | ... | 0.16 $N_2$ |
| S-590 | A | 0.45 | 1.22 | 0.011 | 0.012 | 0.38 | 20.50 | 20.20 | Balance | 19.90 | 4.60 | 4.40 | ... | 4.18 | ... | ...... |
| | V | 0.43 | 1.21 | ... | ... | 0.28 | 20.74 | 19.94 | Balance | 19.71 | 4.47 | 3.81 | ... | 4.20 | ... | ...... |
| V-36 | A | 0.30 | 1.03 | 0.010 | 0.011 | 0.20 | 24.80 | 20.60 | 1.75 | Balance | 4.50 | 2.26 | ... | 1.71 | ... | ...... |
| | V | 0.27 | 0.86 | 0.013 | 0.017 | 0.18 | 24.70 | 19.29 | 1.87 | Balance | 4.39 | 2.31 | ... | 1.71 | ... | ...... |
| WF-31 | A | 0.15 | 1.39 | ... | ... | 0.62 | 20.20 | 9.50 | 3.55 | Balance | 3.10 | 11.32 | 0.59 | ... | ... | Nil Cu |
| | V | 0.156 | 1.42 | 0.009 | 0.016 | 0.42 | 20.34 | 9.93 | 4.13 | Balance | 2.61 | 10.69 | 1.03 | ... | ... | 0.08 Cu |
| L 605 | A | 0.09 | 1.40 | ... | ... | 0.48 | 19.70 | 10.80 | 1.95 | Balance | ... | 16.0 | ... | ... | ... | |
| | V | 0.08 | 1.64 | 0.010 | 0.006 | 0.61 | 21.11 | 10.84 | 1.60 | Balance | ... | 15.54 | ... | ... | ... | 0.03 $N_2$ |

[1]—Allison laboratory.
[2]—Vendor conformance report data.

mens for making short-time elevated temperature tension tests.

Many turbojet engine parts and components operate under conditions of high stress and nonuniform temperature distribution. Since an extended test period was undesirable, preliminary work with the apparatus indicated that static loading of specimens to about 50 per cent of their short-time tensile strength at the temperature of test was productive of results capable of being checked and, therefore, interpretation. A short-time tension test was made on each material just prior to the thermal-shock test for each temperature of interest.

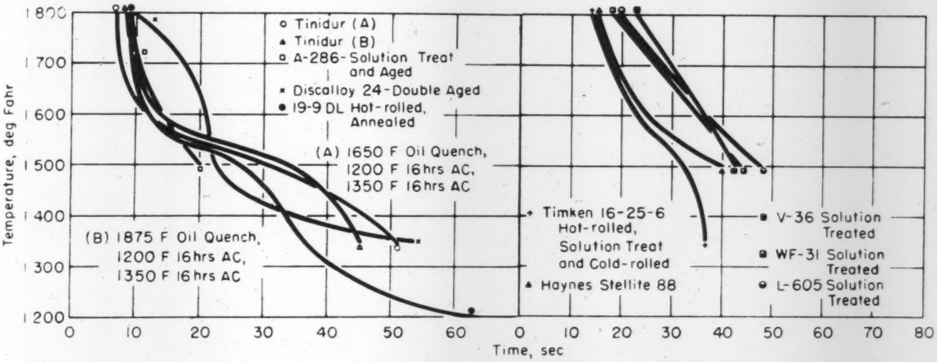


FIG. 5.—Stress Required to Produce Failure in Approximately 100 Cycles for Longitudinal Specimens

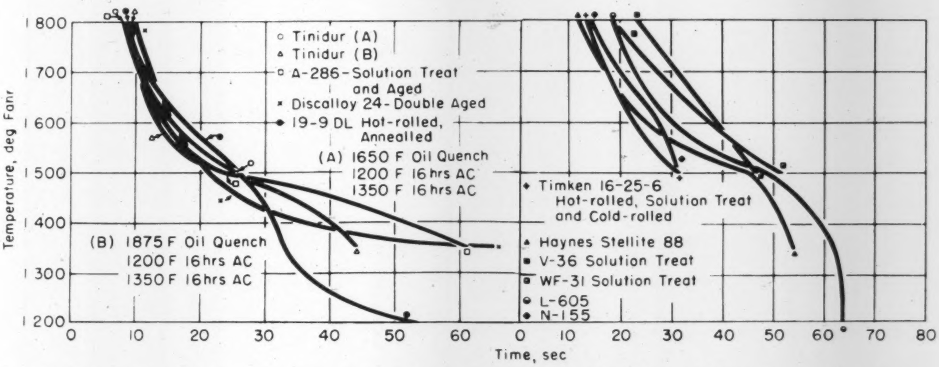
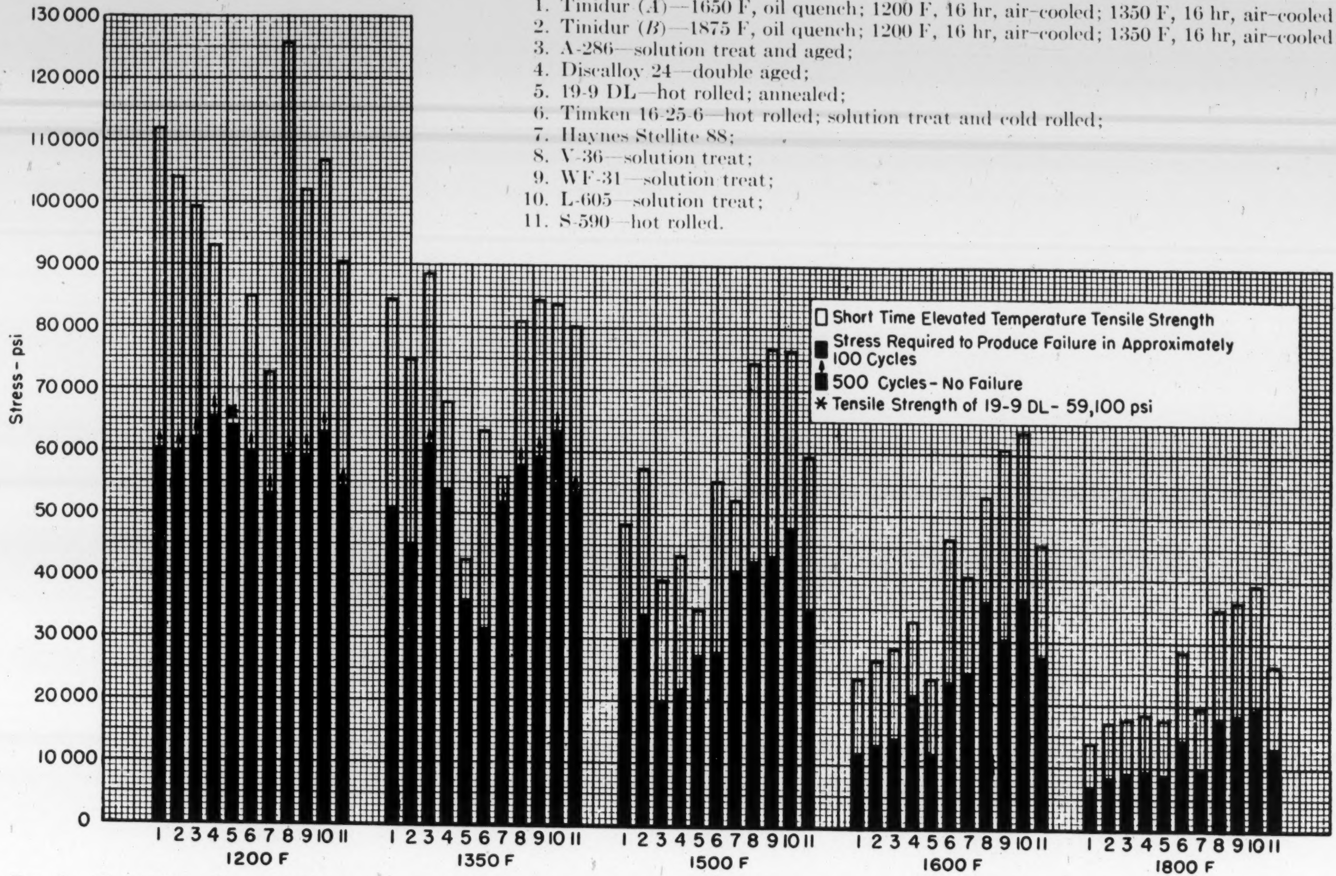


FIG. 6.—Stress Required to Produce Failure in Approximately 100 Cycles for Transverse Specimens.

*Presentation of Data:*

The data obtained in the conduct of this investigation have been plotted in two different forms. The first method, as shown in Figs. 5, and 6, consisted of plotting a calculated stress to produce failure in thermal cycling test under stress in approximately 100 cycles *versus* temperature. This method of plotting was used for data obtained from specimens tested with the axis parallel to the rolling direction and those tested perpendicular (cross-grain) to the rolling direction.

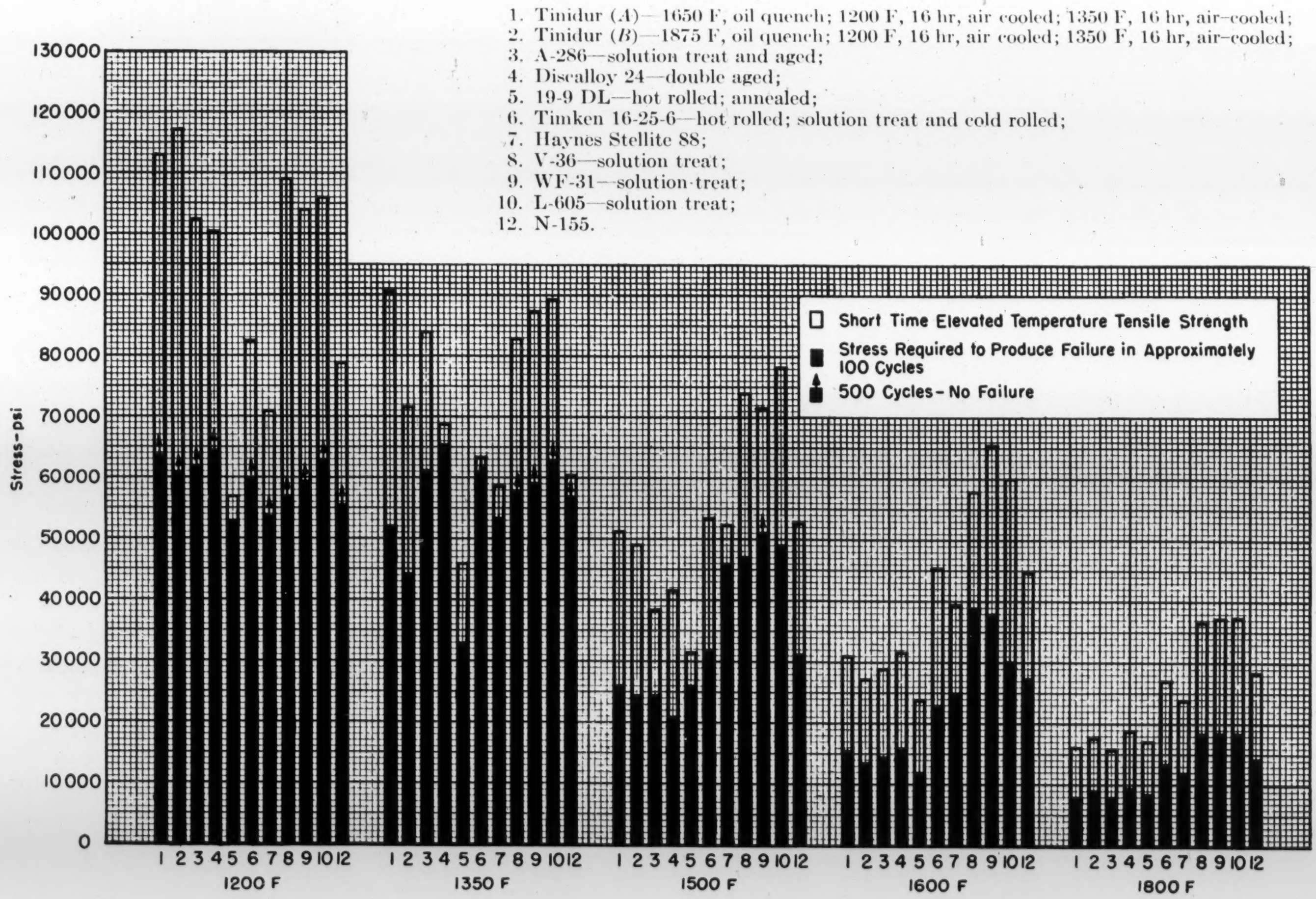
The initial test procedure involved the following:



1. Tinidur (*A*)—1650 F, oil quench; 1200 F, 16 hr, air-cooled; 1350 F, 16 hr, air-cooled;
2. Tinidur (*B*)—1875 F, oil quench; 1200 F, 16 hr, air-cooled; 1350 F, 16 hr, air-cooled;
3. A-286—solution treat and aged;
4. Discalloy 24—double aged;
5. 19-9 DL—hot rolled; annealed;
6. Timken 16-25-6—hot rolled; solution treat and cold rolled;
7. Haynes Stellite 88;
8. V-36—solution treat;
9. WF-31—solution treat;
10. L-605—solution treat;
11. S-590—hot rolled.

Stress - psi
130 000
120 000
110 000
100 000
90 000
80 000
70 000
60 000
50 000
40 000
30 000
20 000
10 000
0
☐ Short Time Elevated Temperature Tensile Strength
■ Stress Required to Produce Failure in Approximately 100 Cycles
■ 500 Cycles - No Failure
* Tensile Strength of 19-9 DL - 59,100 psi
1 2 3 4 5 6 7 8 9 10 11
1200 F
1 2 3 4 5 6 7 8 9 10 11
1350 F
1 2 3 4 5 6 7 8 9 10 11
1500 F
1 2 3 4 5 6 7 8 9 10 11
1600 F
1 2 3 4 5 6 7 8 9 10 11
1800 F

FIG. 7.—Short-Time Elevated Temperature Tensile Strength and Thermal Shock Resistance of Various High-Temperature Sheet Materials Cut Parallel to Grain.



1. Tinidur (*A*)—1650 F, oil quench; 1200 F, 16 hr, air cooled; 1350 F, 16 hr, air-cooled;
2. Tinidur (*B*)—1875 F, oil quench; 1200 F, 16 hr, air cooled; 1350 F, 16 hr, air-cooled;
3. A-286—solution treat and aged;
4. Discalloy 24—double aged;
5. 19-9 DL—hot rolled; annealed;
6. Timken 16-25-6—hot rolled; solution treat and cold rolled;
7. Haynes Stellite 88;
8. V-36—solution treat;
9. WF-31—solution treat;
10. L-605—solution treat;
12. N-155.

☐ Short Time Elevated Temperature Tensile Strength
■ Stress Required to Produce Failure in Approximately 100 Cycles
▲■ 500 Cycles - No Failure
Stress-psi
130 000
120 000
110 000
100 000
90 000
80 000
70 000
60 000
50 000
40 000
30 000
20 000
10 000
0
1 2 3 4 5 6 7 8 9 10 12
1200 F
1 2 3 4 5 6 7 8 9 10 12
1350 F
1 2 3 4 5 6 7 8 9 10 12
1500 F
1 2 3 4 5 6 7 8 9 10 12
1600 F
1 2 3 4 5 6 7 8 9 10 12
1800 F

FIG. 8.—Short-Time Elevated Temperature Tensile Strength and Thermal Shock Resistance of Various High-Temperature Sheet Materials Cut Across Grain.

1. For the first 200 cycles of testing, the stress applied to the specimen was equivalent to 50 per cent of the ultimate tensile strength at the test temperature.

2. For each additional 100 cycles, the stress was increased at increments of 5000 psi until failure occurred. If no failure was obtained, testing was discontinued at 500 cycles.

This method of testing was used through approximately one half of the program.

It was then decided to adjust the procedure as follows:

1. For the initial 200 cycles, same as before.

2. For each additional 100 cycles, the stress was increased by adding a load equivalent to $\frac{1}{3}$ the difference between the existing load and the maximum load possible with the particular equipment. As before, testing was discontinued at 500 cycles. An automatic recorder was an integral part of the test equipment, by means of which an accurate record of the number of cycles of testing was maintained.

Several generalizations can be made from the graphs:

1. The cobalt-base alloys exhibit strengths higher than most of the iron-base alloys throughout the entire range of temperatures used.

2. The iron-base alloys exhibited properties which are very similar throughout the range of temperatures tested. This fact was so pronounced that it was difficult to present the data in graphical form because the points tended to fall within a narrow band at each of the test temperatures. For presentation and clarity, it was necessary to offset some of the points and indicate their true positions by means of arrows. This leaves the logical conclusion that the austenitic iron-base alloys exhibit thermal stress properties that are very similar throughout the range of temperatures 1200 to

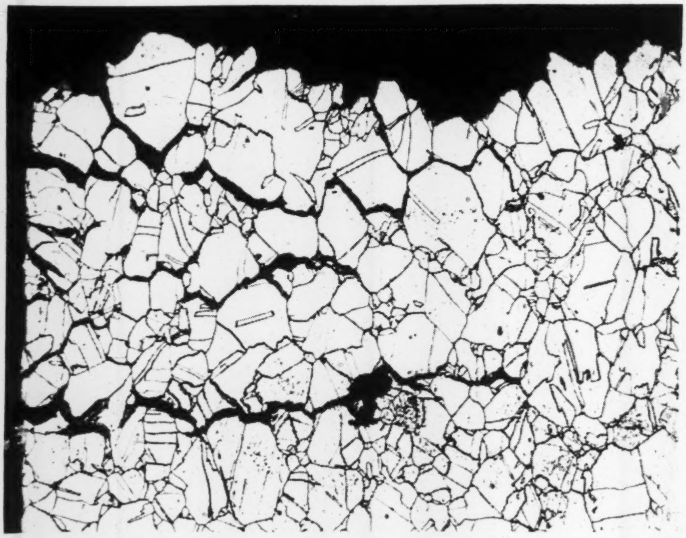


FIG. 9—Alloy: Timken 16-25-6 Tested at 1600 F in Allison GMC Thermal Shock Machine to Failure; Etchant: Marble's Reagent (× 100).

1800 F, and the choice of any particular alloy would depend on other design or fabricating factors.

3. At 1200 and 1300 F it was not possible to calculate a stress which would produce failure at approximately 100 cycles for many of the alloys. None of the alloys tested exhibited poor thermal shock properties under stress at temperatures of 1200 and 1350 F.

Figure 7 presents the data for the material tested in the parallel direction, while Fig. 8 presents that for the cross-direction material.

This type of plotting indicates there is a definite relationship between the stress required to produce failure in approximately 100 cycles and the short-time elevated temperature tensile strength for all of the materials tested. This can be seen by observing that the cobalt-base alloys exhibit exceptionally high tensile strengths and correspondingly high thermal stress values, particularly above 1500 F. This same relation appears to be true of the iron-base alloys throughout the entire range of temperatures tested, and the general statement can be made that the higher the short-time elevated temperature tensile strength the greater will be the stress required to produce failure in thermal shock for any of the alloys tested in this investigation.

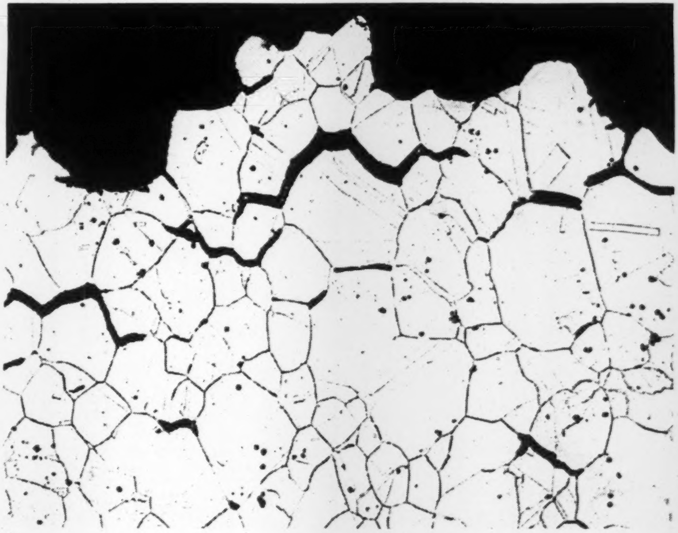


FIG. 10.—Alloy: Discalloy 24 Tested at 1600 F Allison GMC Thermal Shock Machine to Failure; Etchant: Chrome Regia (× 100).

The second method of plotting the data consisted of preparing bar graphs relating the stress required to produce failure in approximately 100 cycles to the short-time elevated tensile strength of the material at the same temperatures. This type of plot was valid for testing at 1500, 1600, and 1800 F, but at 1200 and 1350 F it was necessary to indicate the stress to which the specimen was being subjected at the cessation of testing: that is, after 500 cycles of testing had been completed with no failure occurring.

*Presentation of Metallographic Studies:*

The fracture surfaces of every failed test specimen were examined metallographically to determine the mode of failure. Fracture initiation in all twelve alloys tested, whether iron-base or cobalt-base types, was the same, namely of an intergranular nature. The fractures have closely simulated many service parts failures. Typical fracture structures are shown in Figs. 9, 10, and 11.

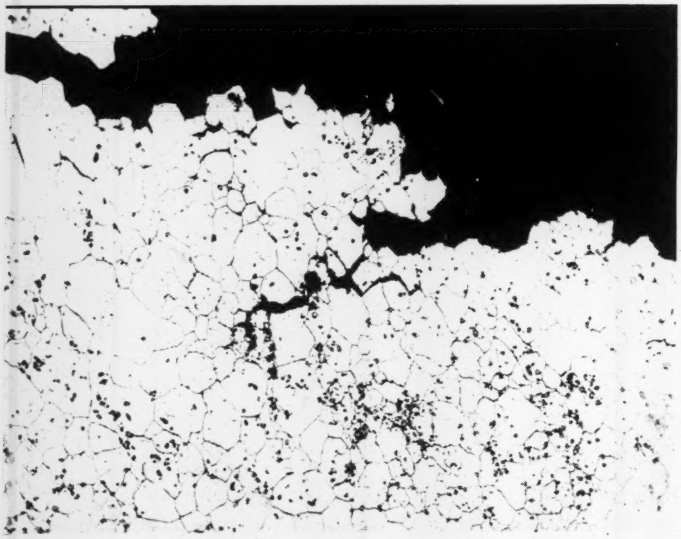


FIG. 11.—Alloy: WF-31 Tested at 1800 F in Allison GMC Thermal Shock Machine to Failure; Etchant: Chrome Regia (× 100).

A conclusion reached as a result of this work is that grain-boundary oxidation is a major reason for deterioration of life expectancy of the high-temperature metals and alloys when used under stress at elevated temperatures. The test program herein reported indicated that the physical properties of the various iron-base and cobalt-base alloys are excellent, particularly at 1200 and 1350 F. It is considered significant that all of the alloys tested also have excellent oxidation resistance at these temperatures. As the temperature of testing approaches 1500, 1600, and 1800 F, there is a marked decrease in physical properties and in the ability of the alloys to resist grain-boundary oxidation. Metallographic evidence is presented in support of this theory.

Figure 12 shows an area of the heated edge of a specimen from the cobalt-base alloy WF-31 after testing at 1350 F for 500 cycles with no failure occurring. It can be seen at the top of the photomicrograph that the grain boundaries show evidence of oxidation.

Figure 13 shows an "internal burst" which was noted *near the heated edge* of a specimen of Timken 16-25-6 alloy after testing at 1200 F. Complete failure had not occurred in 500 cycles of testing, but it is apparent that failure was occurring due to grain-boundary separation. This was the result of tensile stress at the temperatures employed. Close study indicates oxidation in the separated grain

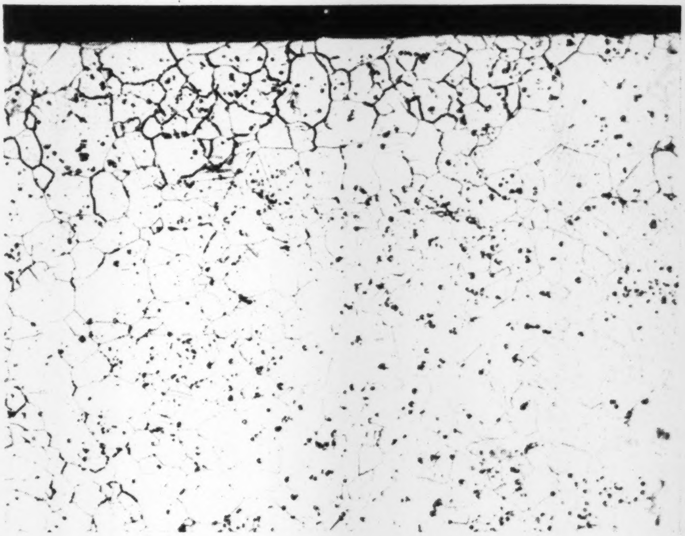


FIG. 12.—Alloy: WF-31 Tested at 1350 F in Allison GMC Thermal Shock Machine; Etchant: Chrome Regia (× 100).

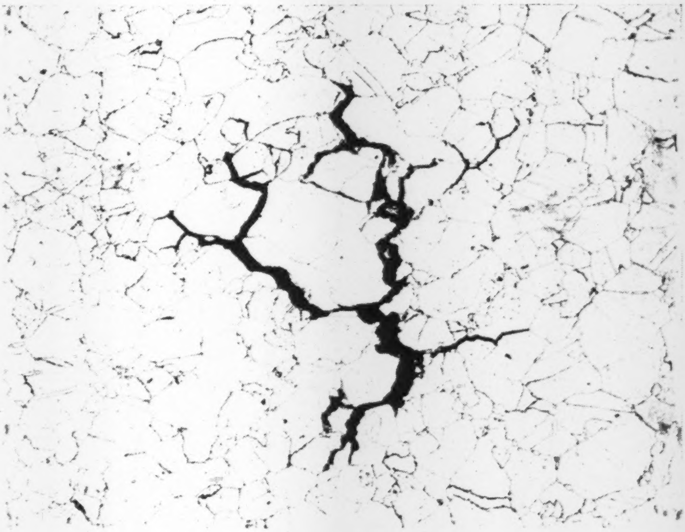


FIG. 13.—Alloy: Timken 16-25-6 Tested at 1200 F, 500 cycles in Allison GMC Thermal Shock Machine; Etchant: Marble's Reagent (× 100).

boundaries which means that surface cracking was in progress.

A review of Figs. 9 and 10 shows grain-boundary oxidation. These are presented in support of a theory that none of the high-temperature alloys in use today is any better than its ability to resist preferential grain-boundary oxidation.

## CONCLUSIONS

1. All of the alloys tested in this investigation exhibited excellent thermal stress properties, especially at 1200 and 1350 F. In some cases a calculated stress corresponding to greater than 90 per cent of the short-time elevated temperature tensile strength would be required to produce failure in approximately 100 cycles.

2. A direct relation exists between stress calculated to produce failure in approximately 100 cycles in thermal shock and the short-time elevated temperature tensile strength of the austenitic high-temperature alloys, namely, the higher the ultimate tensile strength any particular alloy possesses, the higher will be the stress required to produce failure in thermal cycling.

3. Metallographic examination of all fracture surfaces indicated failure to occur by intergranular propagation when the austenitic high-temperature alloys are tested in thermal shock under stress.

4. Metallographic evidence is presented in support of a suggested theory that preferential grain-boundary oxidation precedes tensile failure when many of the high-temperature materials are used in applications involving temperatures exceeding 1200 F. The inherent long-time strength of any alloy for elevated temperature use cannot be realized if the alloys do not also possess resistance to preferential grain-boundary oxidation

# ON DYNAMIC CREEP WITH SPECIAL CONSIDERATION OF STRAIN RATE EFFECTS

By F. H. Vitovec[1]

## Synopsis

The dynamic creep behavior of a number of materials is discussed and classified. The creep properties under simultaneous action of static stress and alternating stress have been derived by several authors from the creep rate-stress relationship under static load. The use of this so derived stress range diagram for creep as a basis for determination of acceleration or retardation of dynamic creep is emphasized. Retardation of creep is attributed to aging effects, and to strain rate effects caused by alternating stress frequency. Acceleration of creep is attributed to metallurgical changes, and overtemperature at very high frequencies. Special consideration is given to the effect of superposition of strain rate and creep rate. The theoretical analysis of the strain rate effects in dynamic creep, presented here, conform with the experimental observation that retardation of creep increases with increasing temperature, and with increasing alternating stress frequency.

The actual service stress in machine parts and structures is generally a combination of static stress and fluctuating stress. Many studies were and are still concerned with the effect of such stress combinations on the rupture properties, and the behavior of a variety of materials has been determined experimentally. The effect of superimposed alternating stress on creep behavior (dynamic creep) has become of increased interest in recent years with the increased use of alloys at elevated temperatures.[2] The design criteria for heat-resistant alloys under simultaneous action of static and alternating stress were primarily based on separate consideration of static creep and reversed stress fatigue. However, dynamic creep depends not only on the operating temperature, static mean stress, type and state of alloy and previous temperature and stress history, but also on amplitude and frequency of alternating stress. The study of the effect of each of these individual variables would not only require the use of a single phase alloy as test material which does not undergo phase changes but also a large number of tests. Due to urgency of the information required, only dynamic creep of relatively complex engineering alloys was studied and the analysis with respect to the effect of the above listed variables could not be performed adequately.

A summary of the published work concerning dynamic creep (**1–14**)[3] is

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[1] Associate Professor, Department of Mechanics and Materials, University of Minnesota, Minneapolis, Minn.

[2] The term elevated temperature is to be understood with respect to the melting temperature of the alloy.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 986.

TABLE I.

| Type of Alloy | Test Temperature, deg Fahr | Alternating to Mean Stress Ratio, $A$ | Test Frequency, cpm | Investigator |
|---|---|---|---|---|
| 0.58 Carbon-Steel | 570, 750, 930 | $\infty$, 1.0 | 50, 500 | **(1)** |
| 0.51 Molybdenum-Steel | 750, 930 | $\infty$, 1.0 | | |
| 15.5 Chromium, 13.3 Nickel 2.02 Tungsten-Steel | 930, 1100 | $\infty$, 1.0 | | |
| Lead | 75 | | | **(2)** |
| 0.58 Carbon-Steel | 930 | Various $0 \leq A \leq \infty$ | 500 | **(3, 4)** |
| 0.51 Molybdenum-Steel | 930 | | | |
| 15.5 Chromium, 13.3 Nickel 2.02 Tungsten-Steel | 930 | | | |
| N-155 | 1350, 1500 | 0, 0.25, 0.67 | 3600 | **(5)** |
| S-816 | 1350, 1500 | | | |
| Vitallium | 1350, 1500 | | | |
| 19-9 DL | 1350 | | | |
| 14S-T Aluminum | 400 | 0.1 | 1200 | **(6)** |
| Lead | 75 | | 3000 | **(7)** |
| 0.26 Carbon-Steel | 750 | $0 \leq A \leq \infty$ | 2000 | **(8)** |
| N-155 | 1350, 1500 | 0, 0.25, 0.67, 1.64, $\infty$ | 3600 | **(9)** |
| S-590 | | | | |
| Vitallium | | | | |
| SAE 4340 | 75, 600, 800, 1000 | 0, 0.2, 0.3, 0.5, 1.0, $\infty$ | 2000 | **(10)** |
| 2024-T4 Aluminum | 300, 400 | 0, 0.15, 0.37, 0.89, 2.16, $\infty$ | 3600 | **(11)** |
| N-155 | 1350, 1500 | 0, 0.25, 0.67 | 11.5, 115 | **(12)** |
| Inconel X | 1350, 1500 | | 3600 | |
| 321 SS | 1500 | | 14,400 | |
| S-816 | 75, 1350 1500, 1650 | 0, 0.25, 0.67 2.0, $\infty$ | 3600 | **(13)** |
| Stellite 31 | 1200, 1500 | 0, 0.25, 0.67 2.0, $\infty$ | 3600 | **(14)** |
| Waspalloy | 1200, 1500 | | | |
| 16-25-6 | 1200 | | | |
| Inconel X-550 | 1500 | 0, 0.25, 1.0 | | |
| Crucible 422 | 900, 1100 | 0.67 | | |
| Lapelloy | 1000 | 0, 0.25, 0.67, 2.0, $\infty$ | | |
| 403 SS | 700, 900 | | | |
| Lead | 90 | $0 \leq A \leq 1$ | 18,000 | **(24)** |

shown in Table I. In this table the type of alloy investigated is listed together with test temperature, ratio of alternating stress to mean stress, and test frequency. At first it appears as if each alloy system displays its own characteristic response to dynamic creep conditions. The analysis of the work listed in Table I is somewhat difficult since no common basis of comparison of dynamic and static creep was used. However, certain features were commonly observed:

(*a*) First and second stage creep occurs under reversed direct stress, that is, under zero static or mean stress.

(*b*) Metallurgical factors play an important role, that is, superimposed alternating stress, primarily of small amplitude, accelerates phase changes and precipitation processes.

(*c*) The first stage creep is larger under dynamic conditions than under static. This is probably due to heat generation caused by damping and time lag of furnace control.

(*d*) The effect of frequency of alternating stress is closely associated with magnitude of stress amplitude, type and state of material, and test temperature.

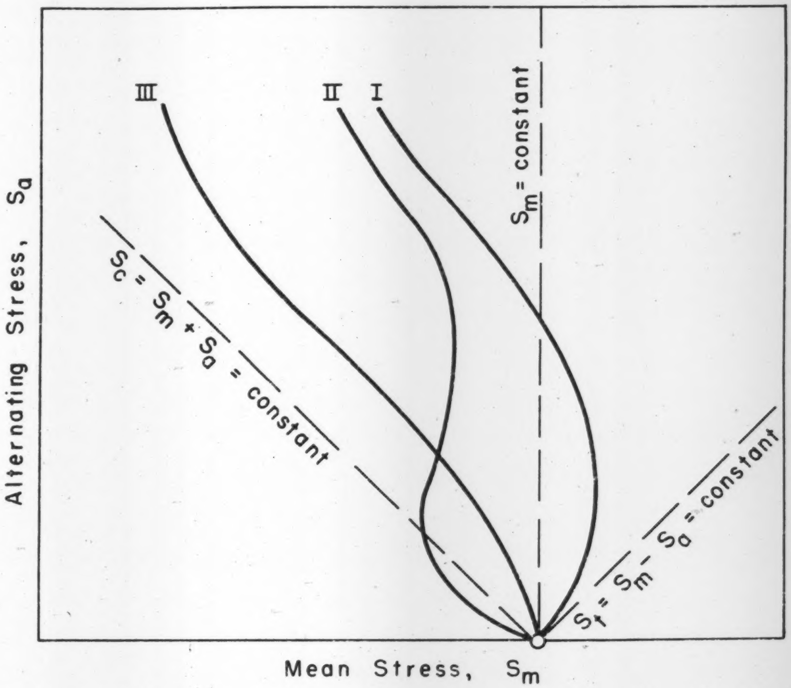
III
II
I
$S_m$ = constant
$S_c = S_m + S_a$ = constant
$S_t = S_m - S_a$ = constant
Alternating Stress, $S_a$
Mean Stress, $S_m$

FIG. 1.—Schematic Stress Range Diagram Showing Curves for Equal Creep in a Certain Constant Time.

If the combinations of alternating and mean stress for constant creep are plotted in a stress range diagram, such as shown in Fig. 1, two basic curves can be observed which classify all materials studied. Curve I is the more frequently observed one, whereas curve II is exhibited by certain alloys only at certain temperatures and frequencies. Curve II apparently indicates the occurence of phase changes due to small amplitude alternating stress. Curves I and II represent the creep behavior only in the frequency range of alternating stress for which the over-all process is isothermal. At very high frequencies the process becomes dominantly adiabatic and creep is accelerated. Also indicated in the diagram of Fig. 1 are lines for constant creep, which are determined by one of the following stresses: maximum stress during cycle, static stress, and minimum stress. The assumption of maximum stress during the cycle as a design criterion results in most cases in considerably smaller stress values, whereas the assumption of static stress as a determining factor results in a too high allowable stress value.

A derivation of creep under combined static and alternating stress was proposed by several authors. These may be briefly reviewed in the following:

A general expression for the relationship of creep rate $u$ and stress $S$ of the form

$$u = u_o \sin h(S/S_o) \qquad \text{(1)}$$

has been discussed by Nadai and McVetty (15), where $u_o$ and $S_o$ are constants. At high stresses this equation becomes

$$u = K_1 S^n \qquad \text{(2)}$$

over a limited range of variables.

The manner in which the stress varies during the test is described by

$$S = S_m + S_a \sin \omega t \qquad \text{(3)}$$

Manjoine (6) determined from Eqs 1 and 3 the ratio of dynamic creep rate $u_d$ and static creep rate $u_s$:

$$\frac{u_d}{u_s} = \frac{1/(2\pi) \int_0^{2\pi} \sin h[(S_m + S_a \sin \theta)/S_o]\, d\theta}{\sin h(S_m/S_o)} = J_o\left(i \frac{S_a}{S_o}\right) \qquad \text{(4)}$$

which is a Bessel function of zero order.

Lazan (5) combined Eqs 2 and 3 and expressed the ratio $E$ of mean dynamic stress to equivalent static stress which will cause the same creep.

$$E = \left[ \frac{t_2 - t_1}{\int_{t_1}^{t_2} (1 + A \sin \omega t)^n\, dt} \right]^{1/n} \qquad \text{(5)}$$

where $A$ is the ratio of stress amplitude $S_a$ to mean stress $S_m$. This equation was solved by graphical methods for exponents $n$ of 7, 9, 11 and presented in the form of a diagram.

Tapsell (8) also used Eqs 2 and 3 for determining the ratios of $S_a$ to $S_m$ causing the same amount of creep. He presented a diagram for rapid evaluation of data for a variety of exponents, $n$, and for both sinusoidal change of stress and stepwise change of stress.

Stress combinations $S_a$ to $S_m$, causing the same amount of creep as determined from Eqs 4 and 5, are shown as curve III in the diagram of Fig. 1. Curve III gives a basis for discussing the effect of alternating stress on creep, since it was derived from the creep behavior under static load and does not consider strain rate effects or metallurgical changes induced by alternating stress. If the experimentally determined curve lies to the left of curve III, creep is accelerated under the particular stress and temperature conditions. If the experimental curve lies to the right of curve III, creep is retarded with respect to static load conditions.[4]

Many of the experimentally determined curves closely follow the theoretical one, curve III, which indicates that in those cases frequency and metallurgical effects are small. Curve I is frequently observed in testing heat-resistant alloys indicating retardation of creep. At small alternating stress amplitudes, curve II exhibits acceleration of creep, whereas at larger stress amplitudes, creep is retarded.

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[4] The terms acceleration and retardation of creep due to superimposed alternating stress are frequently used in the literature without consideration of this basis of comparison.

As mentioned before, the deviation from the theoretical curve III is apparently due to two major factors: phase changes initiated or accelerated by alternating stress, and frequency effects.

Many of the alloys used today are of the precipitation hardening type. For elevated temperature service, these alloys operate in temperature ranges of the aging temperature and above. Metallurgical changes such as continued aging, overaging, and recrystallization due to temperature and static stress are already expressed in the static creep curves and are therefore considered in the theoretical analysis. However, superimposed alternating stress generally accelerates reactions in the solid state. Acceleration of aging, precipitation, and segregation were experimentally demonstrated for aluminum alloys (16) and steel (17, 18). Effects of this kind can either increase or decrease creep rate and total creep depending upon the initial state of the material and the temperature. Frequency is also a factor to be considered when analyzing the effect of alternating stress on solid state reactions. Associated with the frequency effect is a strain rate effect which will be discussed in the next section.

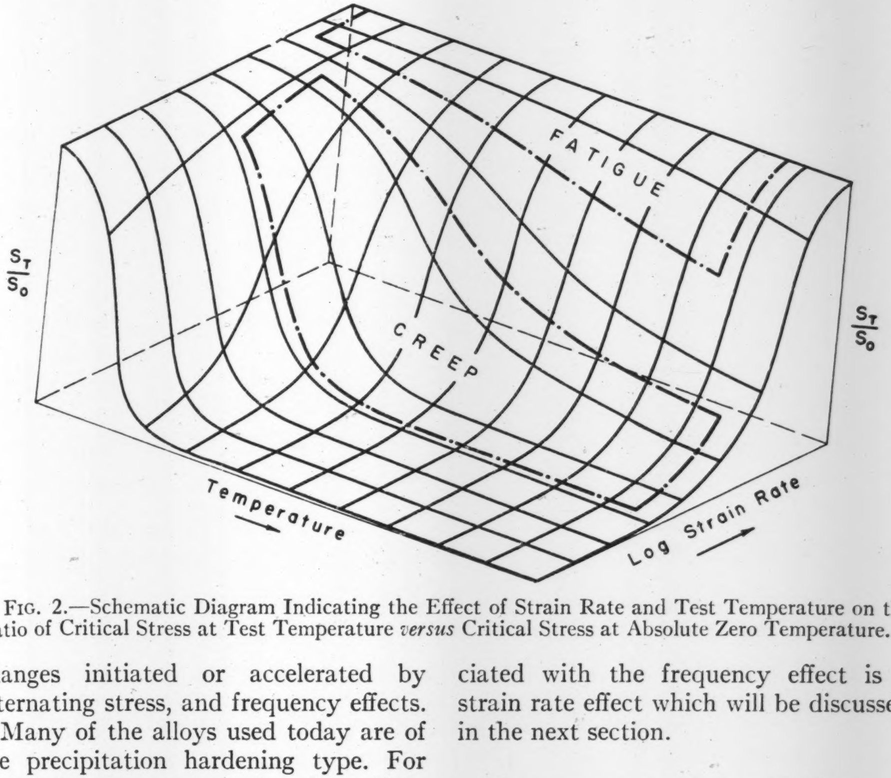

FIG. 2.—Schematic Diagram Indicating the Effect of Strain Rate and Test Temperature on the Ratio of Critical Stress at Test Temperature *versus* Critical Stress at Absolute Zero Temperature.

## SUPERPOSITION OF STRAIN RATE AND CREEP RATE

Of the many variables affecting dynamic creep, the effect of variable strain rate due to alternating stress on the creep behavior of the material plays an important part.

In order to illustrate the effect of temperature and strain rate on critical stress (fatigue strength, creep strength etc.), a schematic diagram derived from the rate theory (21) is shown in Fig. 2. The surface indicated by the heavy lines shows the range of strain rates usually occuring in fatigue and creep tests and the temperature range usually studied. The diagram indicates the effect of temperature on creep which occurs at small strain rate and also illustrates the relatively small effect of temperature on reversed stress fatigue due to the relatively high strain rates at test frequencies. The diagram also shows the increased significance of strain rate at elevated temperature. At any temperature the critical stress is increased with increasing strain rate.

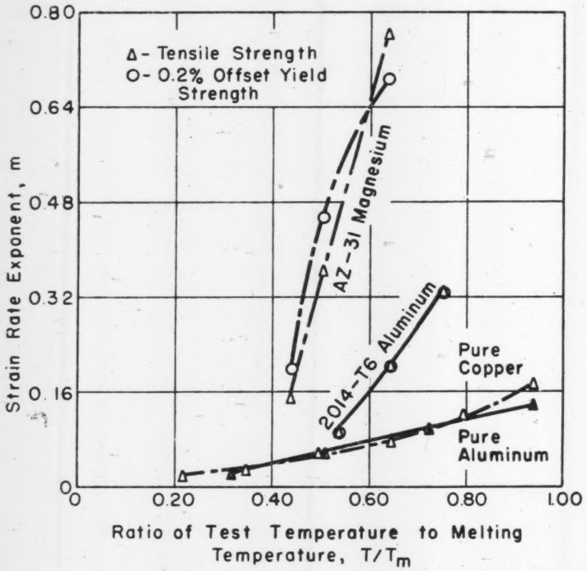

FIG. 3.—Effect of Temperature on the Strain Rate Exponent $m$.

For consideration of the effect of strain rate due to alternating stress on the creep rate due to the static mean stress, it may be assumed that a certain stress exists at which creep initiates. It is very likely that this critical stress varies with strain rate and temperature as illustrated in Fig. 2. Since the critical stress decreases with increasing temperature at a fixed strain rate, creep increases at a fixed dynamic strain rate, while increasing strain rate at constant temperature increases the critical stress and therefore reduces the creep.

Formulas for critical stress as a function of both temperature and strain rate were proposed by several authors (19,20,21). These formulas would have the advantage that metallurgical changes could be expressed by changes of the activation energy. However, in order to arrive at a closed mathematical solution, the simplified expression for strain rate effects on critical stress

$$S = K_2(\dot{\epsilon})^m \ldots\ldots\ldots\ldots (6)$$

is used in the following analysis. Comparison of this formula with Fig. 2 shows that the constants $K_2$ and $m$ are functions of temperature. Figure 3 shows a diagram indicating the variation of the strain rate exponent, $m$, with temperature for various materials (22,23).

The creep rate due to dynamic loading, $u_d$, can be expressed by combination of Eqs 2, 3, and 6 in the following form:

$$u_d = K_1 S_m{}^n (1 + A \sin \omega t)^n - K_1 \left[ K_2 \left( \frac{A S_m \omega}{E} \right)^m \cos^m \omega t \right]^n \ldots (7)$$

The first term expresses the creep rate under variable stress derived from static creep. The second term expresses the reduction of creep rate due to the strain rate caused by alternating stress. The validity of this assumption is confirmed by the experimental data as will be shown later. Integration of Eq 7 within the limits 0 and $2\pi/\omega$ results in the creep strain per cycle $\epsilon_c$ :

$$\epsilon_c = \frac{2\pi}{\omega} K_1 S_m{}^n \sum_{r_1}^{r_2} \binom{n}{2r} \cdot A^{2r} \frac{1 \cdot 3 \cdot 5 \cdots (2r - 1)}{2 \cdot 4 \cdot 6 \cdots 2r} - \frac{2\sqrt{\pi}}{\omega^{1-mn}} \cdot K_1 K_2{}^n \left( \frac{A S_m}{E} \right)^{mn} \frac{\Gamma\left( \frac{mn + 1}{2} \right)}{\Gamma\left( \frac{mn + 2}{2} \right)} . (8)$$

The limits of the summation are from $r_1 = 0$ to $r_2 = (n - 1)/2$ for odd numbers of $n$, and from $r_1 = 0$ to $r_2 = n/2$ for even numbers of $n$. This relationship holds true for even values of $n$ only as long as the alternating stress $S_a$ is smaller or equal to the mean stress $S_m$ , otherwise positive and negative creep must be considered separately. For evaluation of Eq 8, the constants $K_1$ and $n$ can be determined from creep tests under static load, whereas $K_2$ and $m$ may be determined from dynamic creep tests for two different alternating to mean stress ratios, $A$. If the numerical indices denote the specific set of data, the formula for $m$ reads as follows:

$$m = \frac{1}{n} \operatorname{Log} \left\{ \frac{\left( \frac{S_{m1}}{S_c} \right)^n \sum_{r_1}^{r_2} \binom{n}{2r} A_1{}^{2r} \binom{2r - 1}{2r} - 1}{\left( \frac{S_{m2}}{S_c} \right)^n \sum_{r_1}^{r_2} \binom{n}{2r} A_2{}^{2r} \binom{2r - 1}{2r} - 1} \right\} \Big/ \operatorname{Log} \left\{ \frac{A_1}{A_2} \frac{S_{m1}}{S_{m2}} \right\} \ldots\ldots\ldots (9)$$

The constants $K_1$ , $n$, $K_2$ , and $m$, listed in Table II, have been determined from experimental data for several materials for 1 per cent creep strain. The values of the strain rate exponent $m$ determined from dynamic creep tests are of the same order of magnitude as those determined by unidirectional tension tests, shown in the diagram of Fig. 3. No check of the temperature effect on the strain rate exponent is possible at present with the available test data. However, some data concerning frequency effect on dynamic creep were reported by L. A. Yerkovich and G. J. Guarnieri (12) for low carbon N-155 at 1500 F and at frequencies of 115 cpm and 3600 cpm. The evaluation of the data for 1 per cent and 2 per cent creep, according to Eqs 8 and 9, resulted in a strain rate exponent $m$ of 0.257 with ±10 per cent variation.

This relatively close agreement of the experimental data indicates that dynamic creep is subjected to strain rate effects apparently by the same mechanism as that occurring under unidirectional loading.

For consideration of dynamic creep as a design criterion, the theoretical curve (curve III, Fig. 1) derived according to Manjoine, Lazan, and Tapsell from the static creep properties expresses the lower allowable stress combinations if metallurgical changes do not occur. With increasing frequency, that is, increasing strain rate, the stresses causing the same creep in a given time increase to an upper critical frequency at which the process is dominantly adiabatic.

Figure 2 also shows that the effect of strain rate increases with increasing temperature. This trend is indicated by dynamic creep data of several heat-resistant alloys.

Stress rupture properties and dynamic creep are apparently affected by strain rate in the same way.

TABLE II.

| Material | Temperature, deg Fahr | $K_1$ | Creep Rate Exponent, $n$ | $K_2$ | Strain Rate Exponent, $m$ |
|---|---|---|---|---|---|
| 16-25-6 | 1200 | $1.56 \times 10^{-17}$ | 9.38 | 57.76 | 0.0903 |
| S-816 | 1350 | $2.92 \times 10^{-11}$ | 6.47 | 92.62 | 0.286 |
| Waspalloy | 1500 | $1.00 \times 10^{-14}$ | 8.39 | 53.85 | 0.278 |

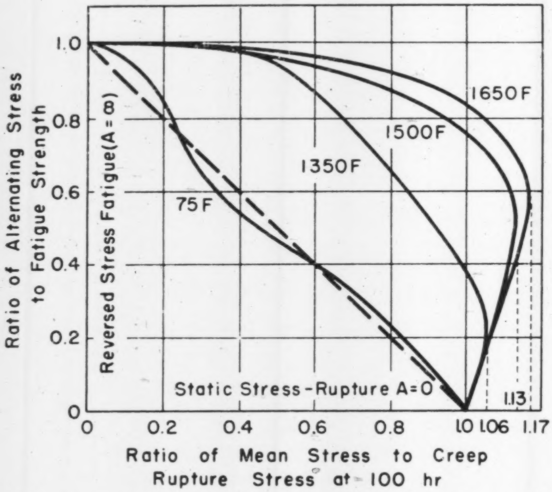

FIG. 4.—Dimensionless Stress Range Diagram for Unnotched Specimens of S-816 Alloy at Temperatures of 75, 1350, 1500, and 1650 F for 100 hr Specimen Life.

Figure 4 shows a dimensionless stress range diagram for stress rupture of S-816 alloy at various test temperatures (13). The tests were performed at a constant frequency of 3600 cpm. As mentioned before, strain rate effects become increasingly significant with increasing temperature. This trend becomes apparent in the diagram of Fig. 4 by the increase in retardation of rupture with increasing temperature.

## CONCLUSIONS

The consideration of dynamic creep as a design criterion appears to be important, since the assumption of maximum stress during a cycle as a creep-determining factor results in considerably smaller allowable stress values, whereas consideration of static creep only results in too high allowable stresses.

Dynamic creep, derived from static creep properties, gives a basis not only for estimating design stress but also for analyzing the effect of alternating stress on the dynamic creep behavior.

Alternating stress superimposed on static stress may initiate or accelerate solid state reactions which may result in either acceleration or retardation of creep depending on the initial state of the material and the type of reaction. The occurrence and the magnitude of this effect depends, in addition to the type and condition of the material, on stress magnitude, temperature, and alternating stress frequency.

Strain rate effects due to alternating stress cause retardation of dynamic creep which increases with increasing test frequency until a maximum occurs which is determined by the transition of the process from isothermal to adiabatic conditions. The mechanism of the strain rate effects on creep strength appears to be the same as that occurring under unidirectional load conditions on other strength properties.

*Acknowledgments*:

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The author wishes to acknowledge the assistance of R. Sack for numerical evaluation of data.

## REFERENCES

(1) M. Hempel and H. E. Tillmanns, "Verhalten des Stahles bei hoeheren Temperaturen unter wechselnder Zugbeanspruchung," (Behavior of Steel at Elevated Temperature under Alternating Tensile Stress), *Mitteilungen des Kaiser-Wilhelm Institutes für Eisenforschung*, Düsseldorf (Germany), Vol. 18, pp. 163–182 (1936).

(2) E. O. Bernhardt and H. Hanemann, "Ueber den Kriechversuch bei dynamischer Belastung und den Begriff der dynamischen Kriechfestigkeit," (On the Creep Mechanism under Dynamic Load and the Definition of Dynamic Creep Strength), *Zeitschrift für Metallkunde,* Vol. 30, pp. 401–403 (1938).

(3) M. Hempel and F. Ardelt, "Verhalten des Stahles in der Waerme unter Zugdruck-Wechselbeanspruchung," (Behavior of Steel at Elevated Temperature under Alternating Direct Stress), *Mitteilungen des Kaiser-Wilhelm Institutes für Eisenforschung*, Düsseldorf (Germany), Vol. 21, pp. 115–132 (1939).

(4) M. Hempel and H. Krug, "Zug-Druck-Dauerversuche an Stahl bei hoeheren Temperaturen und ihre Auswertung nach verschiedenen Verfahre," (Reversed Direct Stress Fatigue Tests with Steel at Elevated Temperatures and their Evaluation by Various Methods), *Mitteilungen des Kaiser-Wilhelm Institutes für Eisenforschung*, Düsseldorf (Germany), Vol. 24, pp. 77–103 (1942).

(5) B. J. Lazan, "Dynamic Creep and Rupture Properties of Temperature-Resistant Materials under Tensile Fatigue Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, pp. 757–787 (1949).

(6) M. J. Manjoine, "Effect of Pulsating Loads on the Creep Characteristics of Aluminum Alloy 14S-T," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, pp. 788–798 (1949).

(7) J. W. Greenwood, "The Influence of Vibration on the Creep of Lead," *Proceedings*, Am. Soc. Testing Mats., Vol. 49 pp. 834–850 (1949).

(8) H. J. Tapsell, P. G. Forrest, and G. R. Tremain, "Creep Due to Pulsating Stresses at Elevated Temperatures," *Engineering*, Vol. 170, pp. 189–190 (1950).

(9) B. J. Lazan and E. Westberg, "Effect of Tensile and Compressive Fatigue Stress on Creep Rupture, and Ductility Properties of Temperature Resistant Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, pp. 837–855 (1952).

(10) W. J. Trapp, "Elevated Temperature Fatigue Properties of SAE 4340 Steel," *TR. 52-325*, Part I, Wright Air Development Center, Dec., 1952.

**(11)** F. W. De Money and B. J. Lazan, "Dynamic Creep, Stress Rupture and Fatigue Properties of 24S-T4 Aluminum at Elevated Temperatures," *TR 53-510*, Wright Air Development Center, Part I, March, 1954.

**(12)** L. A. Yerkovich and G. J. Guarnieri, "Effect of Cyclic Load Frequency on the Creep-Rupture and Fatigue Properties of Jet-Engine Materials," *TR. 55-226*, Wright Air Development Center, June, 1955.

**(13)** F. H. Vitovec and B. J. Lazan, "Creep, Rupture and Notch Sensitivity Properties of S-816 Alloy up to 1650 F under Various Combinations of Fatigue and Static Stress," Symposium on Metallic Materials for Service at Temperatures above 1600 F, Am. Soc. Testing Mats., p. 69–88 (1955). (Issued as Separate publication *ASTM STP No. 174*.)

**(14)** F. H. Vitovec and B. J. Lazan, "Fatigue, Creep and Rupture Properties of Heat Resistant Materials," *TR. 56-181*, Wright Air Development Center, Apr., 1956.

**(15)** A. Naday and P. G. McVetty, "Hyperbolic Sine Chart for Estimating Working Stresses of Alloys at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 735 (1943).

**(16)** R. F. Hanstock, "The Effect of Vibration on a Precipitation Hardening Aluminum Alloy," *Journal*, Inst. of Metals, p. 469 (1948).

**(17)** H. R. Sanders and M. Hempel, "Metallographische und roentgenographische Untersuchungen von Weicheisen nach Kaltverformung und Wechselbelastung," (Metallographic and X-ray Study of Iron after Cold Deformation and Fatigue), *Archiv. für Eisenhuettenwesen*, Vol. 23, pp. 383–405 (1952).

**(18)** H. Schenk and E. Schmidtmann, "Einfluss von Wechselbeanspruchung auf Diffusions- und Ausscheidungsvorgaenge in unlegierten Staehlen," (Effect of Cyclic Stressing upon Diffusion in Plain Carbon Steels), *Archiv. für Eisenhüttenwesen*, Vol. 25, pp. 579–583 (1954).

**(19)** E. Orowan, "Zur Kristallplastizität," (On Crystal Plasticity), *Zeitschrift für Physik*, Vol. 89, pp. 605–633 (1934).

**(20)** C. Zener and J. H. Hollomon, "Effect of Strain Rate Upon Plastic Flow of Steel," *Journal Applied Physics*, No. 15, pp. 22–33 (1944).

**(21)** F. H. Vitovec, "Ueber den Einfluss von Temperatur und Verformungsgeschwindigkeit auf die kritische Schubspannung," (On the Effect of Temperature and Strain Rate on Critical Stress), *Zeitschrift für Metallkunde*, Vol. 43, pp. 128–130 (1952).

**(22)** A. Naday and M. J. Manjoine, "High-Speed Tension Tests at Elevated Temperatures, Parts 2 and 3," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 63, p. A-77 (1941).

**(23)** C. L. Dotson and J. R. Kattus, "Tensile Properties of Aircraft Structural Metals at Various Rates of Loading after Rapid Heating," *Technical Report 55-199*, Wright Air Development Center, Aug., 1955.

**(24)** A. J. Kennedy, "Effect of Fatigue Stresses on Creep and Recovery," International Conference on Fatigue of Metals, London 1956, Session 4, Paper 10 (1956).

# SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON DETERMINATION OF GASES IN METALS

The papers and discussions in this Symposium were presented at the Seventh Session of the Sixtieth Annual Meeting of the American Society for Testing Materials, held in Atlantic City, N. J., June 18, 1957. It was sponsored by Committee E-3 on Chemical Analysis of Metals, with F. M. Fowler acting as Symposium Chairman and presiding over the session.

The papers included in the Symposium are:

Introduction—F. M. Fowler
Two Apparati for the Determination of Gases in Metals—D. L. Guernsey and R. H. Franklin
Application of Vacuum Fusion to Gas-Metal Studies—W. G. Guldner and A. L. Beach
Oxygen Determinations Using a Platinum Bath and Capillary Trap—W. G. Smiley
Bromination-Carbon Reduction Method for the Determination of Oxygen in Metals—M. Codell, G. Norwitz, and Silve Kallmann
Emission Spectrometric Determination of Oxygen in Metals—V. A. Fassel, W. A. Gordon, and R. W. Tabeling

These papers with discussion were issued as *ASTM Special Technical Publication No. 222*, entitled "Symposium on Determination of Gases in Metals."

# MEASURING SHRINKAGE OF CONCRETE BLOCK—A COMPARISON OF TEST METHODS*

By E. L. Saxer[1] and H. T. Toennies[2]

## Synopsis

Concrete masonry units, made with five different aggregates and cured by low-pressure steam or by autoclaving, were tested for drying shrinkage by three different methods. Auxiliary tests were made on prisms cut from companion block. Physical properties of the block and the aggregate were determined.

The data indicate that the Modified British Test on whole block units could be substituted as an accelerated test method of measuring concrete masonry shrinkage as it occurs during normal atmospheric drying. Shrinkage measurements by the Modified British Method required approximately two weeks for completion.

Shrinkage measurements by the accelerated Rapid Test were substantially greater than those occurring with normal drying. No single correlation, applicable to all block, could be established between Rapid Test measurements and normal drying shrinkage, but a fair correlation was noted for block manufactured by a given curing technique.

Shrinkage measurements on cut prism sections were inconsistent and showed poor correlation with results obtained on whole block.

The occasional cracking of walls constructed of concrete masonry has long been regarded as one of the most troublesome problems confronting the concrete products industry.

Obviously the cracks form because stresses are developed which are greater than the strength of the material. These stresses may be brought about by excessive loads, by inadequate foundations, by faulty workmanship, or by restraint of units which are attempting to decrease in volume.

In common with many materials, concrete masonry shrinks when it is cooled or when it dries out. If such shrinkage is resisted by external forces, tension stresses will develop internally with the result that the block may crack. The present consensus is that the shrinkage associated with moisture loss is a significant factor in the development of wall cracks.

In an effort to reduce the probability of shrinkage cracking, some regulatory agencies have adopted specifications limiting the permissible volume change in block conditioned successively at two widely different moisture contents.

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* Presented at The Sixtieth Annual Meeting of the Society, June 16-21, 1957.

[1] Research Foundation, University of Toledo, Toledo, Ohio.

[2] National Concrete Masonry Assn., Chicago, Ill.

Unfortunately there is no universal agreement on the conditions under which such volume changes shall be determined. Generally speaking, all test procedures call for length and weight determinations to be made after test specimens have been immersed in water. (Immersion times vary from 24 to 96 hr.) After the wet measurements have been made, the block are dried under one of several conditions until length and weight equilibrium presumably have been established. The dry measurements are then made and the per cent volume change is calculated. The procedure may call for the drying to be conducted under temperature and relative humidity limitations approximating field conditions, or may require an acceleration of the process by raising the temperature and/or lowering the relative humidity.

Each of the suggested drying methods possesses advantages and disadvantages. Of the three methods which appear to have received the most attention the Reference Test simulates natural drying most closely. It requires block to be dried to equilibrium at a constant temperature and relative humidity, representative of the mean exposure to which exterior and interior masonry walls will be subjected during their service life. Air maintained at 73 F and 50 per cent relative humidity satisfies this requirement, for the two wall types, in most regions of the United States. The chief objection to this type of drying is that as much as 60 days may be required before equilibrium is established. This, of course, is undesirable from the standpoint of acceptance testing, and limits the method's application to research studies.

Committee 716, of the American Concrete Inst. has proposed that high pressure steam cured[3] masonry units be dried at 225 F in a ventilated oven. (Relative humidity is not specified.) This Rapid Test brings about equilibrium in a much shorter time than drying as outlined for the Reference Method. However, there is the objection that the volume changes, thus determined, usually are substantially greater than those determined by methods calling for drying at more nearly normal temperatures.

An intermediate procedure has been adapted from the British Standard Method which calls for drying at 122 F over a saturated solution of calcium chloride. This does not subject the test units to as severe drying conditions as the Rapid Method, but a greater length of time is required to reach an equilibrium condition.

Although much information on volume change-moisture content relations has been obtained in recent years, there are relatively limited data available for direct comparisons of shrinkage measurements made on concrete masonry units dried by each of the methods outlined above. This is particularly true for low-pressure steam-cured[4] block, which constitute the major portion of present-day production.

In order to develop additional pertinent data, a test program was initiated to study the reliability of the various accelerated testing procedures when used on whole block, and on prisms cut from block, cured by either low-pressure or high-pressure steam.

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[3] High-pressure steam cure (HP), or autoclaved, refers to curing of concrete masonry units by live steam at moderate pressures, usually 125 to 150 psi. The two terms are used interchangeably in this report.

[4] Low-pressure steam curing (LP) refers to the curing of concrete masonry units with live steam at atmospheric pressure. The technique is sometimes, more correctly, described as atmospheric steam curing since the curing chamber is under no relative pressures.

This program was sponsored by the National Concrete Masonry Assn., and was carried out in the laboratories of the Research Foundation of the University of Toledo during the summer and fall of 1955.

TABLE I.—AVERAGE MEASURED SHRINKAGE VALUES AND SCATTER BETWEEN MAXIMUM AND MINIMUM MEASUREMENT FOR THREE TEST METHODS.

| Aggregate Type | Cure | Reference Method | | Modified British Method | | Rapid Method | |
|---|---|---|---|---|---|---|---|
| | | Average Shrinkage, per cent | Scatter[a] | Average Shrinkage, per cent | Scatter[a] | Average Shrinkage, per cent | Scatter[a] |
| WHOLE BLOCK SPECIMENS | | | | | | | |
| Expanded slag | HP<br>LP | 0.031<br>0.061 | 0.004<br>0.006 | 0.029<br>0.059 | 0.006<br>0.005 | 0.052<br>0.079 | 0.010<br>0.009 |
| Expanded clay | HP<br>LP | 0.027<br>0.064 | 0.003<br>0.006 | 0.026<br>0.054 | 0.001<br>0.005 | 0.047<br>0.073 | 0.001<br>0.010 |
| Sand and gravel | HP<br>LP | 0.015<br>0.027 | 0.005<br>0.001 | 0.020<br>0.026 | 0.002<br>0.002 | 0.029<br>0.038 | 0.006<br>0.004 |
| Pumice | HP<br>LP | 0.040<br>0.072 | 0.006<br>0.004 | 0.033<br>0.059 | 0.001<br>0.007 | 0.076<br>0.117 | 0.009<br>0.011 |
| Cinders | HP<br>LP | 0.016<br>0.048 | 0.008<br>0.003 | 0.020<br>0.040 | 0.001<br>0.004 | 0.046<br>0.062 | 0.005<br>0.008 |
| Average scatter[a] | ........ | ... | 0.005 | ... | 0.003 | ... | 0.007 |
| CUT PRISM SPECIMENS | | | | | | | |
| Expanded slag | HP<br>LP | 0.022<br>0.045 | 0.005<br>0.005 | 0.017<br>0.045 | 0.011<br>0.007 | 0.045<br>0.070 | 0.008<br>0.004 |
| Sand and gravel | HP<br>LP | 0.022<br>0.036 | 0.007<br>0.016 | 0.008<br>0.033 | 0.011<br>0.011 | 0.012<br>0.038 | 0.028<br>0.017 |

[a] Range in per cent between maximum and minimum measured shrinkage values in group of five test specimens.

## TEST PROGRAM

Tests units for this program were standard 4 by 8 by 16 in. hollow concrete block obtained from cooperating commercial block plants having facilities available for both high- and low-pressure steam curing, and whose normal production included block of the desired aggregate type. The five aggregate types represented were expanded slag, sand and gravel, expanded clay, pumice, and steam-boiler cinders.

Test data on the various aggregates, curing schedules, and pertinent data concerning the test units are given in Appendix I. With the exceptions of the pumice and the low-pressure steam-cured cinder and expanded clay block, the mix proportions and curing procedures were those ordinarily used by the plants in question.

Block were conditioned in the laboratory at 73 F and 50 per cent relative humidity for 10 days preceding tests. During this period plugs for length change measurements were installed in whole block test specimens. Also, in the cases of the expanded slag and the sand and gravel units, a prism for each of the test methods was sawed from each of five block and made ready for test.

Testing began with a 48-hr water immersion period of all block at an age of 28 days. Saturated length and weight measurements were then taken, following which the units were separated into three groups for drying by the Reference, Modified British, and Rapid Methods.

Rapid drying was done by holding the block for 48 hr in a mechanically ventilated oven at 220 to 235 F. After cooling, the specimens were weighed and measured. Because drying was sometimes incomplete in 48 hr, the units were then returned to the oven for an additional 24 to 48 hr of drying, after which they were again cooled and measured.

TABLE II.—COMPARISON OF MEASURED SHRINKAGE MEASUREMENTS BY ACCELERATED TEST METHODS WITH REFERENCE SHRINKAGES.

| Aggregate Type | Cure | Ratio—British Test Value to Reference Shrinkage, per cent | Ratio—Rapid Test Value to Reference Shrinkage, per cent |
|---|---|---|---|
| Expanded slag | HP | 94 | 168 |
| | LP | 97 | 130 |
| Expanded clay | HP | 96 | 174 |
| | LP | 84 | 114 |
| Sand and gravel | HP | 133 | 193 |
| | LP | 96 | 141 |
| Pumice | HP | 83 | 190 |
| | LP | 82 | 163 |
| Cinders | HP | 125 | 287 |
| | LP | 83 | 129 |
| Average | | 97 | 169 |

Modified British drying was accomplished in a cabinet held at 122 F. Air in the cabinet was circulated over a saturated solution of calcium chloride giving a relative humidity of 17 per cent. After three days initial drying, the specimens were cooled in a desiccator to 73 F and measured. Drying was then continued in 24 hr periods until an equilibrium length and weight was reached.

Reference drying was done in an atmosphere maintained constant at 73 F and 50 per cent relative humidity. Weight and length measurements were taken at weekly intervals until specimens had reached an equilibrium condition.

A detailed description of the three test procedures and the equipment used is given in Appendix II.

## RESULTS AND DISCUSSION

The average measured shrinkage values for whole block and cut prism specimens tested by each of the three methods are presented in Table I. In all cases, the values shown are the averages of tests on five specimens. In an effort to portray the degree of consistency for the various tests, the range between the maximum and minimum values for each series is tabulated under the heading "Scatter." Values shown in these tables are length changes in terms of percentage of the specimen's saturated length (10 in. for whole block, 6 in. for prisms).

Measured shrinkages by the two accelerated drying methods are compared with corresponding Reference values in Table II. As may be noted, the Modified British values were from 82 to 133 per cent of the Reference measurements, with an average ratio, for the ten samples, of 97 per cent. The percentage difference between the two test methods (Reference and Modified British) is less than 10 in four cases and below 20 in eight. Rapid test measurements showed a much larger variation. They ranged from 14 to 187 per cent greater (ratios of 114 to 287 per cent) than the corresponding Reference values, having an average ratio of 169 per cent.

These data (Table II) indicate that the Modified British test could be used as an accelerated method of measuring concrete masonry drying shrinkage. Modified British results were substantially the same as the shrinkages due to normal drying (Reference test). It is equally apparent that results by the Rapid test could not be used, *per se*, but that some corrective factor would have to be applied to them.

If the average Rapid to Reference ratio of 1.69 were applied to each of the Rapid test measurements listed in Table I, the corrected values would still deviate from normal drying shrinkages by too great a margin for acceptability. The corrected values (not shown) would range from 67 to 170 per cent of the corresponding Reference shrinkages. Therefore, it appears that several different corrective factors, which take into account differences other than test procedure, would have to be determined and applied before the Rapid test could be satisfactorily used.

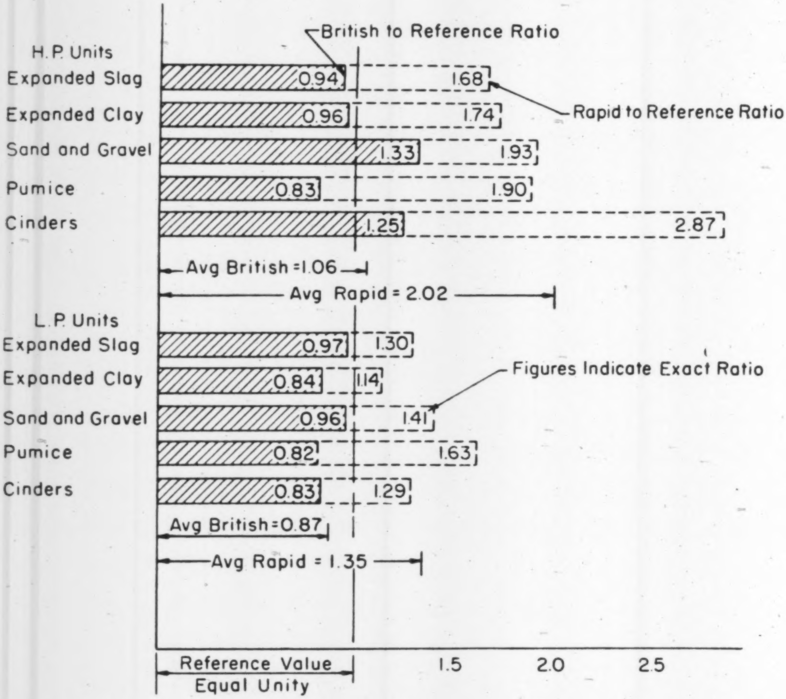

FIG. 1.—Comparison of Measured Shrinkage Values by the Modified British and Rapid Test Methods with Those Obtained by the Reference Test.

One possible solution is demonstrated in Fig. 1. Here the shrinkage ratios presented in Table II have been plotted in two separate groups according to the curing technique employed in block manufacture. The individual Rapid/Reference ratios are grouped somewhat closer to the average as computed for a single curing technique. Applying the two different averages (low-pressure cure = 1.35, high-pressure cure = 2.02) to the individual Rapid measurements of Table I gave the following corrected values:

| | Low-Pressure Cured Units (Factor = 1.35) | | High-Pressure Cured Units (Factor = 2.02) | |
|---|---|---|---|---|
| Aggregate | Corrected Rapid value, per cent shrinkage | Ratio, corrected value to Reference, per cent | Corrected Rapid value, per cent shrinkage | Ratio, corrected value to Reference, per cent |
| Expanded slag | 0.058 | 95 | 0.026 | 84 |
| Expanded clay | 0.054 | 84 | 0.023 | 85 |
| Sand and gravel | 0.028 | 104 | 0.014 | 93 |
| Pumice | 0.087 | 120 | 0.038 | 95 |
| Cinders | 0.046 | 96 | 0.023 | 144 |

As may be noted, the corrected values, thus determined, are all within 20 per cent of their Reference shrinkages, except for those of the high-pressure steam-cured cinder block.

The tests on cut prisms were made in an effort to learn if there is a consistent relationship between shrinkages of whole block and small sections. If such a relationship were to exist, the problems of testing laboratories would be considerably simplified because smaller and less elaborate drying chambers could be used. Unfortunately the data obtained during this program showed no consistent relationship. It also appeared that prisms yield results having much greater scatter than whole block. Prism shrinkages compared to those of companion whole block were as follows:

| Test Method | Ratio of Prism to Whole Block Shrinkage, Range |
|---|---|
| Reference | 0.71 to 1.47 |
| Modified British | 0.40 to 1.27 |
| Rapid | 0.41 to 1.00 |

Based upon these data, it would appear that there is reason to doubt the reliability of shrinkage measurements made on cut prisms. It is worth noting that in all cases the prisms were saturated and dried side by side with the whole block.

In marked contrast to the prisms, the measurements on whole block showed rather small values of scatter. The average deviation for all three test methods was less than 0.0002 in. (0.002 per cent). It is possible that because of equipment variations and personnel factors there would be less uniformity if test results from a number of different laboratories were compared. However, it seems significant that one operator, using the same equipment throughout, obtained much more uniform results on measurements of whole block than on cut prism specimens. Insofar as the three test methods are concerned, when using whole block, there appears to be no appreciable difference in the magnitude of scatter. While not indicated in the tables, it should be mentioned that warping of whole block was insignificant in all cases.

Although the idea of tests on small specimens is attractive from the standpoint of ease in handling, as well as simplification of equipment needs, it presents some problems in the laboratory technique involved and some theoretical objections. It is possible, for instance, that moisture will leave the sawed surfaces of a cut specimen at a different rate than from the original molded surfaces. Furthermore, since there is only one gage point on each end of a prism specimen there is no way to evaluate warping effects. From a practical standpoint, the chief objections are that some of the materials are extremely hard and difficult to saw and drill to close tolerances, while others are so soft as to be quite fragile when reduced to a 1 by 1 by 6-in. prism.

The accelerated shrinkage tests can be further evaluated by comparing the shrinkage ratios of high-pressure to low-pressure steam-cured block, as measured by the various tests. It is known that high-pressure steam-cured concrete masonry units normally undergo less volume change during drying than low-pressure steam-cured units of like aggregate. Therefore, an acceptable accelerated shrinkage test should give about the same shrinkage ratio between the two type block as the Reference test. Calculated ratios are listed in Table III. It is important to note that the measured reduction in shrinkage which accompanies high-pressure steam curing is greatest when units are tested by the Reference Method, less by the Modified British, and is least evident when the units are dried in the Rapid oven. The adoption of the Rapid Method as a standard acceptance test would be unfair to the producer using autoclave or equivalent curing unless suitable corrective factors were established and applied.

TABLE III.—RATIO OF MEASURED SHRINKAGE FOR HIGH PRESSURE CURED TO LOW PRESSURE CURED UNITS BY THE VARIOUS TEST METHODS.

| Aggregate Type | Ratio—Shrinkage of High Pressure to Low Pressure Steam Cured Block | | |
|---|---|---|---|
| | Reference Test | British Test | Rapid Test |
| Expanded slag | 0.51 | 0.49 | 0.66 |
| Expanded clay | 0.42 | 0.48 | 0.64 |
| Sand and gravel | 0.56 | 0.77 | 0.76 |
| Pumice | 0.56 | 0.56 | 0.65 |
| Cinders | 0.33 | 0.50 | 0.74 |
| Average | 0.47 | 0.56 | 0.69 |

The advantage of the Rapid Method is that test results would be available in a comparatively short time. It has been rather widely accepted that 48 hr of drying at 225 F will suffice to bring block to an equilibrium condition. However, unpublished data obtained in various investigations at the University of Toledo suggested that this assumption may, in many cases, be in error insofar as either block weight or length is concerned. Therefore, in the work covered by this report, all Rapid Method block were dried 48 hr at 220 to 235 F, cooled, measured, and then replaced in the oven for an additional 24 to 48 hr of drying. In the cases of the sand and gravel and expanded clay units there was no measurable difference in the results at 48 and 72 hr of drying. For the remaining aggregates there was a considerable increase in shrinkage as the drying time was extended. Additional shrinkage values for these aggregates were as follows:

| Aggregate | Cure | Total Measured Shrinkage, per cent | |
|---|---|---|---|
| | | 48 Hr Drying | Additional Drying |
| Expanded slag | HP | 0.025 | 0.052 |
| | LP | 0.063 | 0.079 |
| Pumice | HP | 0.070 | 0.076 |
| | LP | 0.105 | 0.114 |
| Cinders | HP | 0.039 | 0.046 |
| | LP | 0.058 | 0.062 |

The Rapid Method shrinkage values listed in Table I are based upon the measurements made after additional drying.

Table IV presents data obtained by oven drying (225 F) the Reference and Modified British test specimens after

TABLE IV.—RESIDUAL SHRINKAGE TO OVEN-DRY CONDITION, MOISTURE CONTENT AND REQUIRED DRYING TIME FOR WHOLE BLOCK SPECIMENS DRIED TO AN EQUILIBRIUM CONDITION BY THE REFERENCE AND MODIFIED BRITISH TEST METHODS.

| Aggregate Type | Cure | Reference Test | | | Modified British Test | | |
|---|---|---|---|---|---|---|---|
| | | Residual Shrinkage, per cent | Residual Moisture, per cent abs. | Drying Time, days | Residual Shrinkage, per cent | Residual Moisture, per cent abs. | Drying[a] Time, days |
| Expanded slag........ | HP | 0.037 | 18.4 | 42 | 0.019 | 11.0 | 7 |
| | LP | 0.042 | 27.9 | 42 | 0.030 | 13.6 | 7 |
| Expanded clay........ | HP | 0.023 | 5.1 | 28 | 0.027 | 2.8 | 6 |
| | LP | 0.025 | 20.5 | 35 | 0.026 | 6.3 | 6 |
| Sand and gravel...... | HP | 0.022 | 12.4 | 28 | 0.009 | 13.7 | 6 |
| | LP | 0.029 | 15.2 | 14 | 0.013 | 11.9 | 6 |
| Pumice.............. | HP | 0.060 | 11.6 | 35 | 0.052 | 5.6 | 7 |
| | LP | 0.077 | 21.8 | 35 | 0.062 | 14.1 | 7 |
| Cinders.............. | HP | 0.026 | 12.2 | 28 | 0.021 | 6.8 | 8 |
| | LP | 0.032 | 24.6 | 28 | 0.026 | 12.7 | 8 |

[a] Drying time for modified British includes only the period units were actually in cabinet.

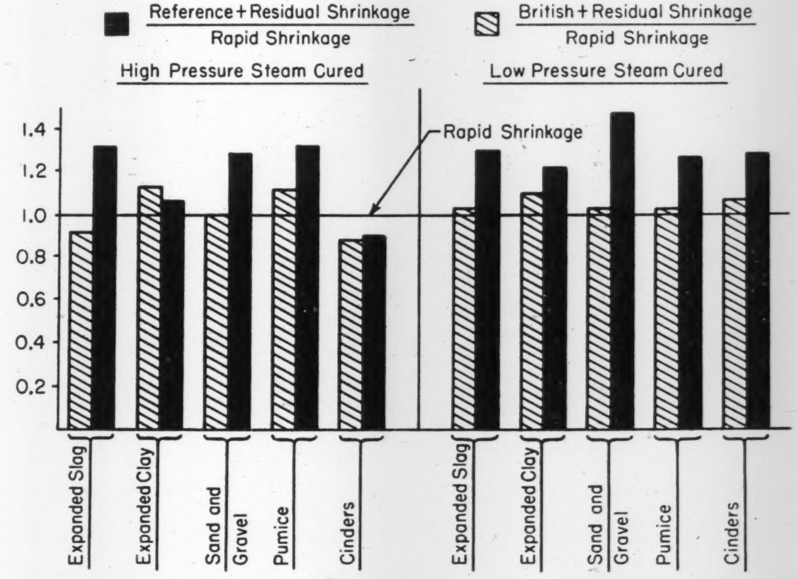

FIG. 2.—Comparison of the Total Shrinkages Obtained by Drying Concrete Block From Saturated to an Oven-Dry Condition by Three Methods.

these units had reached an equilibrium condition at 73 and 122 F respectively. The extra shrinkage and moisture loss resulting from this process is termed residual. Examination of this table shows that all units underwent further length and weight decreases when subjected to the higher Rapid-oven drying temperature. The table also shows that, except for the autoclaved sand and gravel units, all block were dried to a lower residual moisture content by the Modified British test than by the Reference Method, even though measured shrinkages as determined by the British Method were less than Reference values for eight of ten samples. It appears that accelerated drying brings about a different moisture loss-shrinkage relationship than normal drying. Possibly shrinkage is to some extent dependent upon a time phenomenon similar to plastic flow.

By combining the values of Tables I and IV the total shrinkage for Reference and Modified British plus residual drying can be determined. Figure 2 presents a comparison which shows that in nearly all cases the rapid values were less than the total of Reference plus residual, while the Modified British shrinkages plus residual values are only slightly less than the Reference plus residual. This again suggests that a time factor, which has little or no effect on shrinkage when accelerated drying methods are used, enters the picture when drying takes place more slowly.

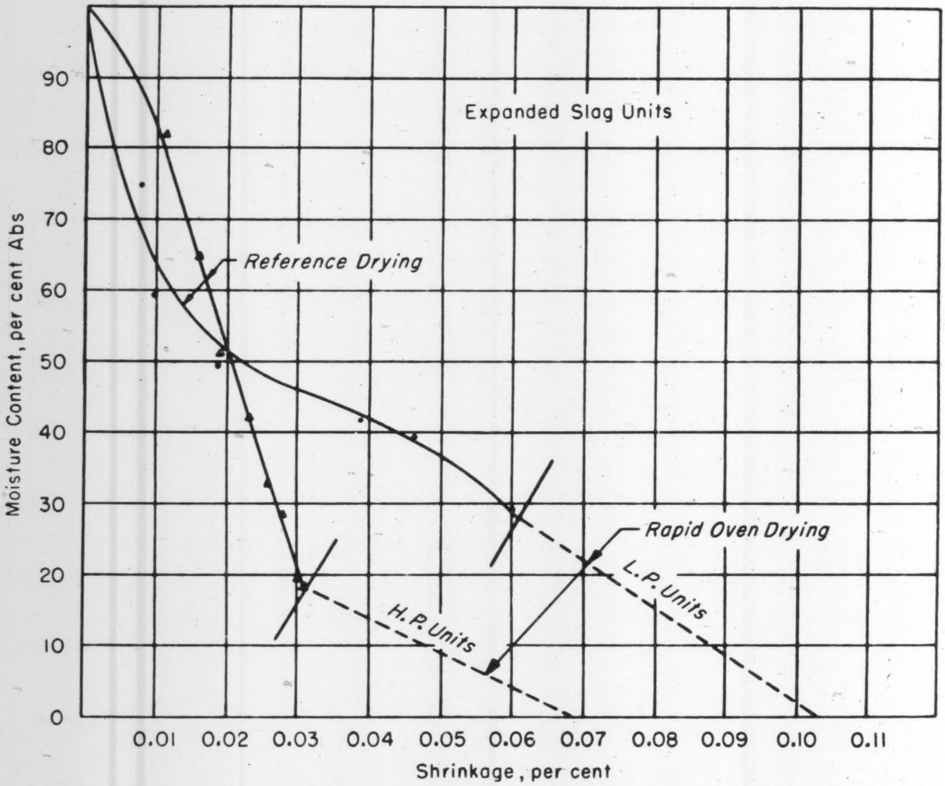

FIG. 3.—Volume Change-Moisture Loss Relationship of 4-in. Hollow Expanded Slag Concrete Block.

Table IV also lists the drying time required to bring about an equilibrium condition in the various aggregate type block by the Reference and Modified British test methods. It should be stated that the times given for the

Modified British Method include only those periods when the block were actually in the drying chamber. If the cooling periods are included, the total time requirement will be from three to five days more than that listed. For specimens tested in this investigation, the total time requirement from water immersion to final dry measurement ranged from five to eight days for the Rapid test, twice that amount for the Modified British, and four to five times that long for the Reference Method.

An explanation as to why a single corrective factor, which would apply to both high-pressure and low-pressure steam-cured block, could not be established for the Rapid tests may be seen in Figs. 3 to 7. These figures give the shrinkage-moisture content curves, for the various aggregate types and curing techniques, when block were dried first to equilibrium by normal Reference drying (solid lines), then placed in the Rapid oven (dashed lines). The moisture loss-shrinkage relationship for block made with the two curing techniques was quite different. Low-pressure steam-cured block shrank at an increasing rate down to a certain moisture content from which point on their shrinkage rate was fairly constant, down to an oven - dry condition. High - pressure steam-cured block generally did not display an increasing shrinkage rate while stored in the Reference drying

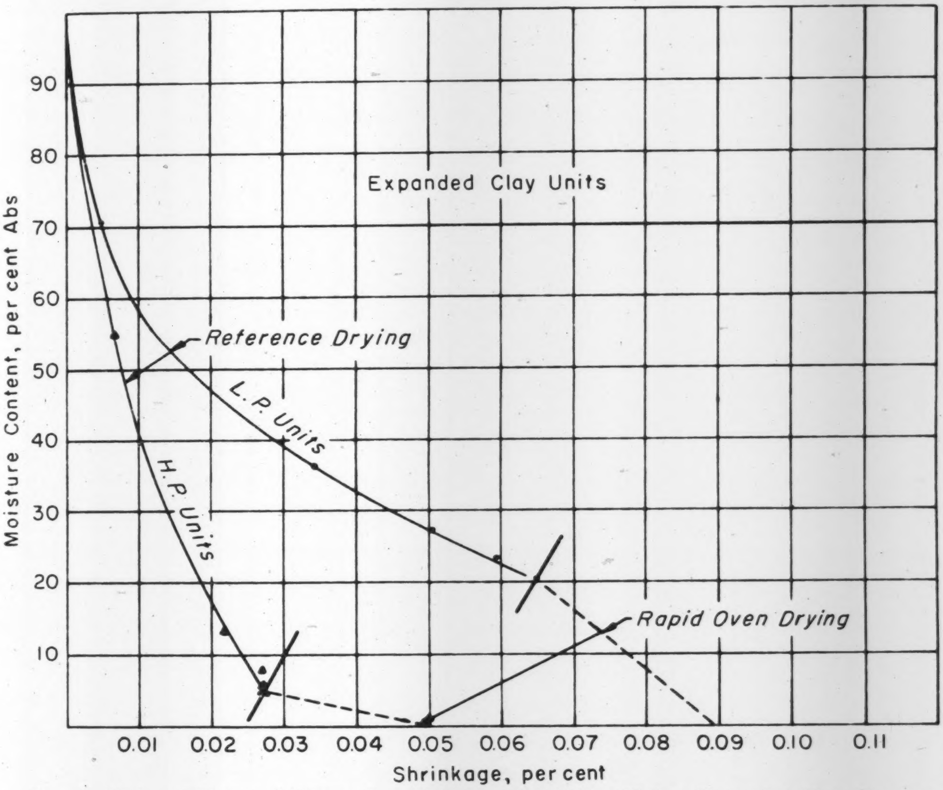

FIG. 4.—Volume Change-Moisture Loss Relationship of 4-in. Hollow Expanded Clay Concrete Block.

chamber (an exception to the above is noted in the case of the high-pressure steam-cured pumice block). However, these units had a very different rate when they were subjected to the Rapid oven, following equilibrium with Reference storage. Evidently their shrinkage rate changes at some moisture content below that of equilibrium in normal Reference storage. Since the change of slope always occurred at some moisture content during Reference drying with the low-pressure steam-cured units but seldom occurred above the Reference Test equilibrium moisture content with high-pressure steam-cured units, no simple correlation could be established between Reference and Rapid drying which would be equally suitable for block made by each of the curing techniques.

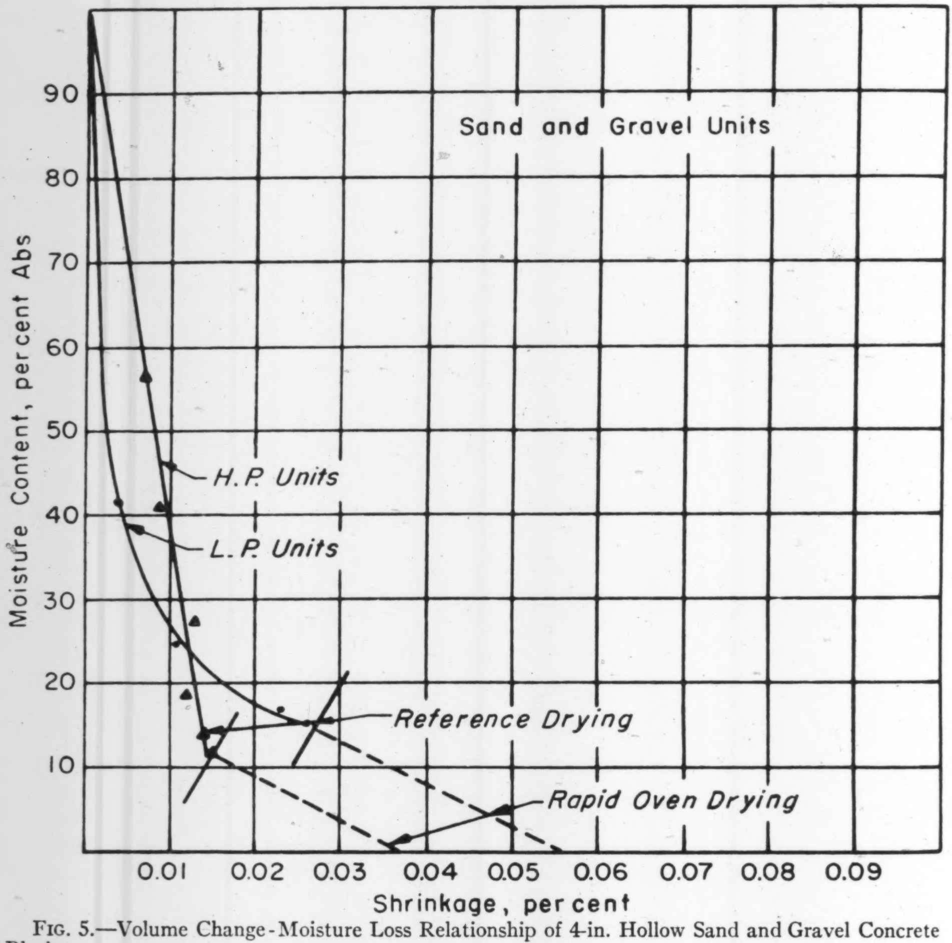

FIG. 5.—Volume Change-Moisture Loss Relationship of 4-in. Hollow Sand and Gravel Concrete Block.

## SUMMARY

1. The Modified British test can be used as an accelerated acceptance test for measuring concrete block shrinkage. Results obtained on whole block specimens, tested by the Modified British

Method, were substantially the same as the shrinkages obtained by Reference drying.

2. Shrinkage measurements obtained by the Rapid test were substantially greater than the shrinkages due to normal drying.

3. No single correlation factor could be established between Rapid and Reference test shrinkages which would be suitable for all block. Correlation between the two tests, for whole block specimens made by a given curing technique, was good for nine out of ten samples.

4. Individual measurements on samples of five whole block specimens had very little scatter, regardless of test method. The average deviation from the mean was less than 0.0002 in.

5. The use of cut prisms in lieu of whole block did not give uniform results; nor did the average shrinkage

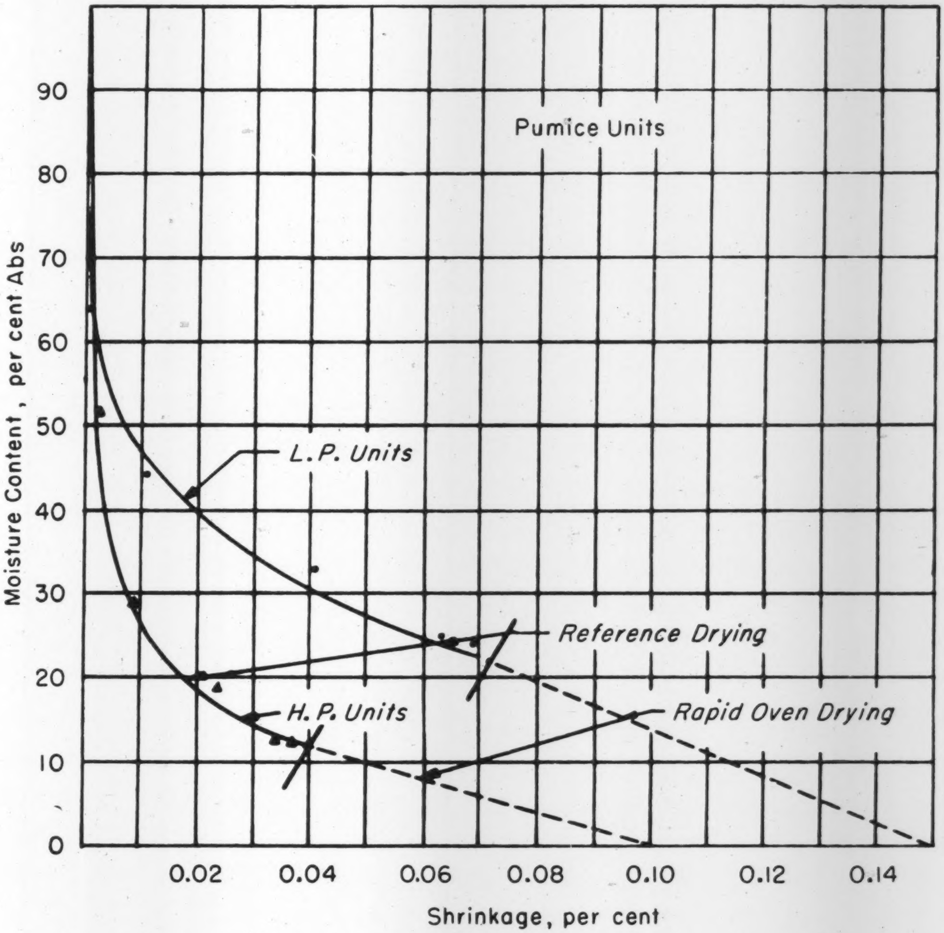

FIG. 6.—Volume Change - Moisture Loss Relationship of 4-in. Hollow Pumice Block.

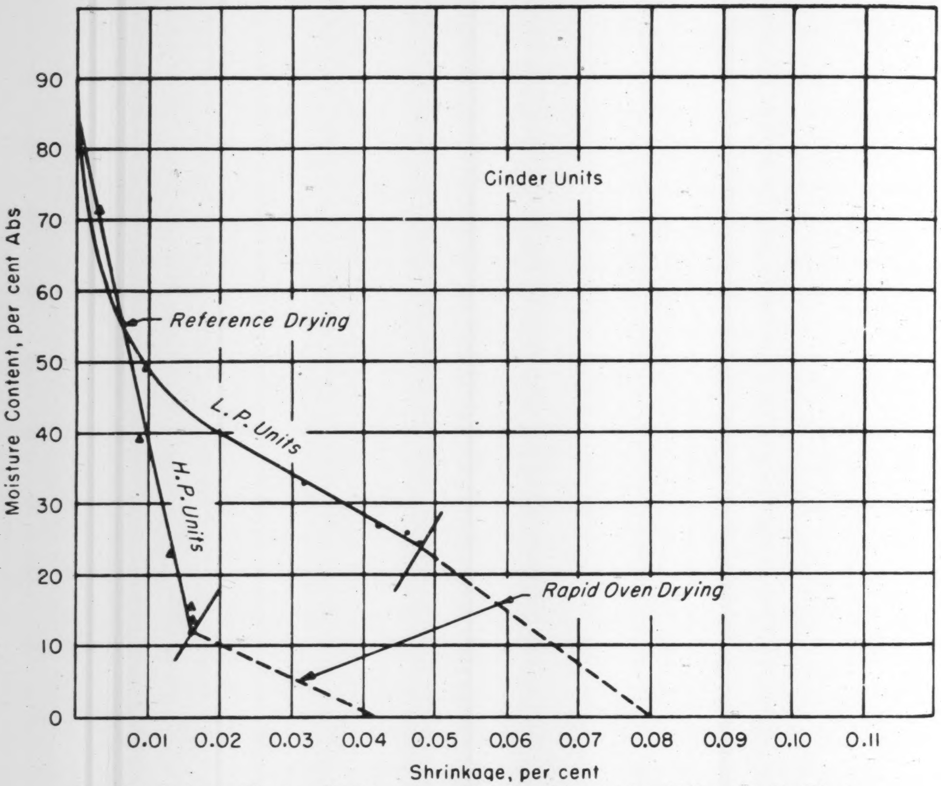

FIG. 7.—Volume Change - Moisture Loss Relationship of 4-in. Hollow Cinder Block.

values thus obtained show a consistent relationship to those from whole block.

6. Forty-eight hours of drying in the Rapid oven at 220 to 235 F was often not enough to bring about an equilibrium condition in block with respect to either weight or length.

*Acknowledgments:*

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## SELECTED BIBLIOGRAPHY ON CONCRETE MASONRY DRYING SHRINKAGE MEASUREMENT

(1) Committee 716, Am. Concrete Inst. (ACI), "Physical Properties of High-pressure Steam-cured Concrete Block," *Journal*, Am. Concrete Inst., Vol. 49, Apr., 1953, p. 745.

(2) H. W. Easterly, "Correlation of Shrinkage and Curing in Concrete Masonry Units," *Journal*, Am. Concrete Inst., Vol. 48, Jan., 1952, p. 393.

(3) Relation of Shrinkage to Moisture Content in Concrete Masonry Units, *Housing Research Paper No. 25*, U. S. Housing and Home Finance Agency.

(4) "Methods for the Determination of Drying Shrinkage and Moisture Movement," British Standard Specification, Precast Concrete Blocks, *B.S. 2028: 1953*, Appendix B, p. 14.

## APPENDIX I

## TEST SPECIMENS USED IN DRYING SHRINKAGE MEASUREMENTS

TABLE V.—PROPERTIES OF AGGREGATES USED IN MANUFACTURE OF TEST SPECIMENS.

| Aggregate Type | Gradation, per cent coarser than: | | | | | | | Fineness Modulus | Specific Gravity, Saturated Surface Dry | Absorption, per cent by weight | Unit Weight, lb per cu ft |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 3/8 | No. 4 | No. 8 | No. 16 | No. 30 | No. 50 | No. 100 | | | | |
| Expanded Slag | | | | | | | | | | | |
| Fine | 0 | 5 | 29 | 46 | 71 | 87 | 96 | 3.34 | 1.71 | 14.8 | 52.4 |
| Coarse | 0 | 87 | 93 | | | | | 5.80 | 1.26 | 17.8 | 39.9 |
| Sand | 0 | 4 | 27 | 42 | 65 | 90 | 98 | 3.26 | 2.62 | 1.2 | 120.4 |
| Combined | 0 | 18 | 39 | 54 | 75 | 90 | 97 | 3.73 | | | 50.0 |
| Expanded Clay | | | | | | | | | | | |
| Fine | 0 | 0 | 10 | 28 | 57 | 71 | 82 | 2.48 | 2.00 | 12.1 | 68.0 |
| Coarse | 0 | 54 | 97 | 99 | | | | 5.50 | 1.84 | 13.9 | 54.0 |
| Combined | 0 | 21 | 43 | 55 | 73 | 82 | 89 | 3.62 | | | 72.0 |
| Sand and Gravel | | | | | | | | | | | |
| Fine | 0 | 2 | 27 | 48 | 77 | 94 | 98 | 3.46 | 2.63 | 1.2 | 118.2 |
| Coarse | 0 | 51 | 91 | 98 | 99 | | | 5.39 | 2.64 | 1.4 | 110.8 |
| Combined | 0 | 18 | 48 | 64 | 84 | 92 | 97 | 4.08 | | | |
| Pumice | 0 | 1 | 18 | 47 | 84 | 92 | 97 | 3.39 | 1.46 | 16.1 | 52.5 |
| Cinders | 0 | 8 | 32 | 51 | 75 | 86 | 92 | 3.44 | 2.15 | 5.2 | 68.5 |

TABLE VI.—COMPOSITION AND RELATED DATA ON 4-IN. HOLLOW CONCRETE BLOCK TEST UNITS.

| Aggregate Type | Cure | Mix, by Weight, (Portland cement Fly Ash) to Aggregate | Yield,[a] blocks per bag | Weight,[b] lb | Absorption, lb per cu ft | Compressive Strength, psi | |
|---|---|---|---|---|---|---|---|
| | | | | | | 1 day | 28 day |
| Expanded slag | HP | 1 to 4.4[c] | 53 | 16.6 | 13.9 | 1700 | 1630 |
| | LP | 1 to 4.4 | 32 | 17.2 | 12.1 | 720 | 1490 |
| Expanded clay | HP | 1 to 6.1 | 49 | 16.3 | 15.3 | 1960 | 2200 |
| | LP | 1 to 5.1 | 41 | 17.5 | 12.5 | 790 | 2670 |
| Sand and gravel | HP | 1 to 9.6[c] | 53 | 26.5 | 7.9 | 3670 | 3460 |
| | LP | 1 to 8.3 | 33 | 26.2 | 7.3 | 1160 | 1830 |
| Pumice | HP | 1 to 3.0 | 27 | 13.5 | 15.4 | 1040 | 1020 |
| | LP | 1 to 3.0 | 27 | 15.0 | 13.5 | 710 | 1050 |
| Cinders | HP | 1 to 5.4[c] | 65 | 18.4 | 15.9 | 1420 | 1540 |
| | LP | 1 to 6.4 | 37 | 19.8 | 13.6 | 880 | 1760 |

[a] 4 by 8 by 16-in. units.

[b] Weight upon completion of curing.

[c] These mixes contained fly ash.

Four-inch hollow concrete block for use in the shrinkage tests were produced at three cooperating block plants having facilities for both autoclave and atmospheric steam curing. These plants were: The Anchor Concrete Products Inc., Buffalo, N. Y., The Cinder Block Inc., Detroit, Mich., and Harter Marblecrete Stone Co. Inc., Oklahoma City, Okla.

In manufacturing the block the standard procedure was to make a batch for one curing method followed immediately by a batch for the second method. Aggregate and cement samples were obtained in the interval between mixes. It is felt that this system minimized extraneous variables by reducing the possibility of variation in aggregate properties and atmospheric conditions. Table V lists the sieve analysis, specific gravity, absorption, and unit weight of each aggregate used.

TABLE VII.—CURING CYCLES USED IN MANUFACTURE OF TEST SPECIMENS.

| Aggregate Type | Preset Period | Steam-up Period | Constant Temperature or Pressure | Soaking Period or Blowdown |
|---|---|---|---|---|
| ATMOSPHERIC STEAM-CURED UNITS | | | | |
| Expanded slag | 4 hr, 80 F | 1¼ hr, 160 F | None | 14 hr, 130 F[a] |
| Expanded clay | 2½ hr, 100 F | 3 hr, 180 F | None | 8 hr, 110 F |
| Sand and gravel | 1¼ hr, 80 F | 2¾ hr, 200 F | None | 2½ hr,[b] 120 F |
| Pumice | 2 hr, 100 F | 4 hr, 180 F | None | 14½ hr, 90 F |
| Cinders | 2 hr, 80 F | 4 hr, 155 F | 4 hr, 155 F | 7 hr, 75 F |
| HIGH-PRESSURE STEAM-CURED UNITS | | | | |
| Expanded slag | 6 hr, 160 F | 3 hr, 140 psi | 5 hr, 140 psi | 45 min. |
| Expanded clay | 2½ hr, 100 F | 3 hr, 145 psi | 5 hr, 145 psi | 35 min. |
| Sand and gravel | 6 hr, 120 F | 3 hr, 145 psi | 5 hr, 145 psi | 45 min. |
| Pumice | 1½ hr, 110 F | 3 hr, 140 psi | 5 hr, 145 psi | 45 min. |
| Cinders | 1½ hr, 100 F | 4 hr, 140 psi | 4 hr, 140 psi | 40 min. |

[a] Minimum temperature at end of soaking period.

[b] Soaking period for atmospheric steam-cured sand and gravel units was stopped after 2½ hr because of an emergency in the plant operation and is not considered adequate or typical of the manufacturer's curing cycle. The block were not discarded but were included in the tests since it was felt that the shortened soaking period would have no adverse effects on their relative shrinkage. Some effect of this shortened soaking period may be observed in the lower than normal compressive strength of sand and gravel block and with low pressure steam (see Table VI).

Mix composition and related data on the test units are given in Table VI. Type IA portland cement was used in all mixes. In the cases of the autoclaved cinder, expanded slag, and sand and gravel block, a portion of the cement was replaced by fly ash in accordance with the normal practice of the respective plants. The yields shown in Table VI have been computed on the basis of block per bag of portland cement only, even though a fly ash substitution was made in some cases. With the exceptions of the pumice units and the low-pressure steam-cured cinder and expanded clay block, the mix proportions used in the manufacture of test units were those ordinarily used by the plant in question.

Upon completion of molding, the test units were held for various preset periods and then subjected to curing cycles as shown in Table VII. Following their cure, the block were marked with identifying symbols, and representative specimens weighed and tested for compressive strength. The remaining units were then crated in stout wooden boxes and, after plant storage of one week, were shipped to the University of Toledo.

## APPENDIX II

## TEST PROCEDURES AND EQUIPMENT USED IN DRYING SHRINKAGE TESTS

### *Pre-Test Storage and Preparation:*

Block for use in all tests were stabilized in the laboratory a minimum of 10 days at 73 F and 50 per cent relative humidity before commencing tests. During this storage period suitable contact plugs for use with the Whittemore strain gage were cemented into each face of whole block test specimens, and prism sections were sawed from the expanded slag and the sand and gravel units. The prisms were cut along the 8-in. dimension of the block in an effort to insure a true representation of the compaction throughout its height. One prism, 6 in. long by 1 in. wide by face shell thickness (about 1 in.) for each test method was sawed from each of five block.

Contact plugs for use with the Whittemore strain gage were cut from $\frac{3}{8}$-in. brass rod. The plugs were cut on a lathe to a length of $\frac{1}{2}$ in.

A drill press equipped with a $\frac{3}{16}$-in. carbide tipped drill was used to sink two holes to a depth of $\frac{5}{8}$ in. in each face of each block. The holes were placed at mid-height of the units and were symetrically spaced at a distance of 10 in.

A neat portland-cement paste was prepared and each hole filled about one third full with the paste. A plug was then forced into the hole until slightly below the face of the block. After a 10-in. gage bar had been used to prick-punch the brass plug, the excess paste squeezed from the hole was wiped off. Upon completion of several days cure, a No. 58 drill was used to sink holes in each plug at the points previously marked. Drilled holes were carried to a depth of $\frac{1}{4}$ in., and any burrs left by the drill were removed by use of a special reaming tool supplied with the Whittemore strain gage kit.

Measuring points for the cut prisms were $\frac{5}{16}$ in. stainless steel ball bearings. A $\frac{1}{4}$-in. drill was used to make a hole $\frac{1}{4}$ in. deep in the center of each end of each prism. These holes were filled with portland-cement paste and ball bearings pressed into the paste so that slightly over half of the ball was embedded. After several days of curing, the ball bearings were carefully cleaned off.

The use of brass plugs was entirely satisfactory. Over 600 plugs were set during the course of this program and only one became loosened during the test. The metal is easy to cut and drill and is hard enough so that a great many readings can be taken without fear of enlarging the hole sufficiently to alter the readings.

There was no difficulty in drilling the holes accurately in the face of the units. It is recommended that a slightly undersize drill be used so as to give a reasonably close fit, and thus keep the cementing material to a minimum.

The sawing of prisms was accomplished with no particular difficulty although some of the specimens were not exactly 1 in. wide. However, with the sand and gravel prisms it was found necessary to use an extremely sharp drill to avoid "chewing" the end of the specimen. Also, some difficulty was experienced with some prisms in locating the hole exactly in the center of some prisms ends.

The portland-cement paste was completely satisfactory for holding the plugs and ball bearings. Only one bearing and one plug were lost throughout the test. However, three prisms were broken by handling while the tests were in progress. In addition, five prisms were spoiled while drilling ball bearing holes and had to be replaced.

### *Test Procedures and Equipment:*

Shrinkage tests were begun 28 days after block manufacture. Step by step procedures were as follows:

1. Thirty block of a given aggregate type (15 autoclaved, 15 atmospheric steam cured) were placed in a water tank held at 73 F by a thermostatically controlled immersion heater.

2. After 48 hr the immersed weight and length of each specimen were determined. Length readings for the block were taken with the units positioned so that the gage line being measured was ⅛ to ¼ in. above the water line.

3. The units were then removed from the tank, held on a ⅜-in. screen for 1 min, lightly toweled off and weighed (all weights to nearest 0.01 lb).

4. Five autoclaved units and an equal number of atmospheric steam-cured specimens were then placed in each of three drying chambers as described below:

(*a*) Reference drying.—A room 8 by 8 by 10 ft, air conditioned with a Kathabar unit, freezer, and heater to maintain a temperature of 73 ± 2 F and a relative humidity of 50 ± 2 per cent. A record of temperature and relative humidity was obtained by automatic recording devices. At no time during the test did the temperature or relative humidity deviate beyond the prescribed limits. Units were stored on 2 by 2-in. wood strips with a spacing of several inches between specimens. Air circulation was maintained by means of a 12-in. fan.

(*b*) Modified British drying.—An insulated, moderately air-tight cabinet having a volume of approximately 10 cu ft. Heat was provided by a series of thermostatically controlled electric strip heaters mounted in the cabinet lid, and separated from the block by a metal baffle. An 8-in. fan circulated the air over the block, the calcium chloride solution, and the heaters. The saturated solution of calcium chloride was placed in pyrex containers on the floor of the cabinet. An open grill of ⅜-in. rods was then positioned over the solution pans and the block were placed on the grill. At least 2 in. of clearance was provided on all sides of each block.

Excess crystals of calcium chloride were provided so as to maintain the solution in a slushy state. When deemed advisable, additional salt was added and excess water removed from the pans. Approximately 25 sq in. of exposed solution area was provided for each cu ft of cabinet volume. This amount of salt dried 10 regular 4 by 8 by 16-in. hollow concrete block (an average total moisture reduction of 2.1 lb per block) to a condition in equilibrium with its own vapor pressure (a relative humidity of 17 per cent at 122 F) in from six to eight days. Cabinet temperature was accurately maintained at 122 ± 2 F.

(*c*) Rapid drying.—An electrically heated, thermostatically controlled, mechanically ventilated oven maintained at 220 to 235 F, and having a volume of approximately 10 cu ft.

5. Actual drying cycles for the three test methods were as follows.

(*a*) Reference Method.—Weight and length of each unit were determined at 1, 3, and 7 days after being placed in the drying room. Thereafter, weight and length measurements were made at weekly intervals until equilibrium was reached. (Equilibrium, as defined for all methods, was attained with an average length change of less than 0.002 per cent since the previous reading, and an average weight change of not more than 0.2 per cent of the last previously determined weight.)

(*b*) Modified British Method.—Test units were first held in the storage cabinet for a 72-hr period, after which they were removed and placed in 55-gal steel drum containing a desiccant of dry calcium chloride and fitted with a gasketed, tight-fitting clamp-on cover. Following cooling to 73 F (usually an overnight process) they were weighed, measured, and replaced in the cabinet for further drying. The second and subsequent drying cycles were of 24 hr duration. The drying, cooling, measuring cycle was continued until an equilibrium condition, as defined above, had been attained.

NOTE.—After attaining an equilibrium status in the Reference and Modified British procedures, test units were placed in an oven (see Rapid test equipment) and held at 220 to 235 F for 48 hr. They were then cooled, weighed, and measured to determine residual values.

(*c*) Rapid Method.—Test specimens were placed in the Rapid oven and held at 220 to 235 F for 48 hr. They were then removed to a cooling chamber, as described for the Modified British Method, and cooled to room temperature. Following weight and length measurements, they were then replaced in the oven for an additional 24 hr of drying. If upon remeasuring they were shown not to have reached an equilibrium condition, the additional 24 hr drying cycle was repeated, which proved sufficient in all cases.

(*d*) Prisms.—In the cases of the expanded slag and the sand and gravel units, a set of prisms was carried through each test method alongside companion whole block.

All length measurements on whole block were made on two gage lines, one on each face of each unit. The mean length change of the two measurements was taken as the unit's shrinkage, to minimize any effects of unit warping. Length changes with the 10-in. Whittemore strain gage were read to the nearest 0.0001 in. Before and after each set of readings, the strain gage was checked against a standard steel reference bar.

Prism length changes were determined by means of a comparator fitted with a dial reading directly to 0.0001 in. All readings were referred to a standard bar. The anvils of the comparator were recessed so as to provide a positive seat for the ball bearings.

All weights were determined on direct-reading Toledo scales accurate to 0.01 lb.

Compression tests were made at 28 days on a 300,000-lb hydraulic testing machine. Testing was in accordance with applicable ASTM standards.

Aggregate absorption and specific gravity tests were made by means of a new technique recently developed at the Research Foundation for use on lightweight aggregates. This test is described in the May, 1956, issue of *Rock Products*.

## DISCUSSION

Mr. S. B. Helms[1] (*presented in written form*).—These comments on the results given in the Saxer-Toennies paper are presented with the thought of comparing some features of the British and Rapid test procedures and raising some questions about the conditions of test with a view to eliminating possible sources of variation between laboratories when using these procedures.

Some individuals have expressed misgivings about oven-drying shrinkage test procedures and have stated 220 F is an excessive test temperature. We undertook a small assignment to determine the effect of oven drying to constant length at 170 F which is approximately midway between the temperatures used in so-called Rapid drying and in the so-called modified British drying.

The accompanying Table VIII includes data for drying to constant length, at successive temperatures of 170 and 216 F, of high-pressure steam-cured block from two of the lots prepared as test specimens for the Am. Concrete Inst., 716 cooperative series. The cinder and lightweight aggregate units are represented by the letters *C* and *L*, respectively, in column at left. Equilibrium shrinkage at 170 F was nearly as great as at the higher temperature. Total "indirect" Rapid shrinkage was found to be only 15 per cent greater, or 0.005 percentage points higher than the shrinkage at 170 F. These data were confirmed by similar tests of cinder block prepared for the same program by another producer.

This points to the fact that the oven temperature effect is a matter of degree, and similar type data could be obtained at an even lower oven drying temperature, approximating the British conditions, which no doubt would be lower than the results at 170 F. The weight change data are shown at the bottom of this table to indicate the specimens had reached constant weight at 170 F. It would seem axiomatic that as the drying temperature is reduced for the purpose of lowering the shrinkage level, the time to reach equilibrium is lengthened.

Table IX shows the agreement between several laboratories which made tests by Rapid procedure on two lots of specimens made up for the Am. Concrete Inst., 716 series. These units were similar to the specimens of Table VIII. It is apparent that all laboratories were in close agreement when this procedure was used and it should be of interest to examine the agreement between laboratories using high-pressure steam-cured blocks from various sources as furnished for the larger cooperative test program.

The deviations reported in Table X resulted from a study of the variability of shrinkage test results for nine large lots of block produced at three locations and tested by four laboratories. Attention is called to the average values for each type of aggregate. In each case the greatest variation occurred using the Reference method and the least variation in Rapid tests, while the results by the British method were intermediate. It will be noted that tests of cinder and sand and gravel block which were manufactured by producer No. 1 showed a high degree of variation when tested by British method. In these particular instances the results by Rapid method had exceptionally low variation.

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[1] Research Engineer, Lehigh Portland Cement Co., Allentown, Pa.

TABLE VIII.—SHRINKAGE TESTS OF HIGH-PRESSURE STEAM-CURED BLOCKS BY OVEN DRYING AT 170 AND 216 F, AVERAGE TEST RESULTS FOR WHOLE BLOCK.

| Specimen | Expansion in Soaking | | Oven Dried at 170 F | | | | Dried at 216 F | | Final Weight, g |
|---|---|---|---|---|---|---|---|---|---|
| | 24 hr | 48 hr | 3 days | 5 days | 7 days | 11 days | 1 day | 2 days | |
| TOTAL LENGTH CHANGE, PER CENT | | | | | | | | | |
| C 1 | 0.014 | 0.012 | 0.039 | 0.037 | 0.036 | 0.035 | ...... | ...... | ...... |
| C 2 | 0.018 | 0.017 | 0.037 | 0.037 | 0.038 | 0.036 | ...... | ...... | ...... |
| L 1 | 0.020 | 0.018 | 0.036 | 0.036 | 0.035 | 0.036 | ...... | ...... | ...... |
| C 3 | 0.012 | 0.017 | 0.033 | 0.033 | 0.033 | 0.033 | 0.036 | 0.036 | ...... |
| L 2 | 0.008 | 0.009 | 0.031 | 0.036 | 0.034 | 0.038 | 0.041 | 0.043 | ...... |
| L 3 | 0.013 | 0.010 | 0.024 | 0.035 | 0.035 | 0.036 | 0.042 | 0.045 | ...... |
| INTERVAL WEIGHT CHANGES, g CORRESPONDING TO ABOVE READINGS | | | | | | | | | |
| C 1 | 1374 | 65 | −1581 | 4 | −8 | +3 | ...... | ...... | 12 981 |
| C 2 | 1412 | 45 | −1600 | 3 | −6 | +2 | ...... | ...... | 13 061 |
| L 1 | 1491 | 120 | −1807 | 1 | −8 | 0 | ...... | ...... | 12 600 |
| C 3 | 1400 | 65 | −1533 | −5 | +2 | −5 | −20 | −4 | 12 965 |
| L 2 | 1580 | 120 | −1757 | −23 | 0 | −2 | −17 | −5 | 12 546 |
| L 3 | 1569 | 105 | −1742 | −19 | 0 | −3 | −18 | −4 | 12 704 |

These variations reveal some uncertainty about agreement between laboratories when making British tests and indicate that cooperative tests should be run to insure that interlaboratory agreement will be satisfactory. It would be a decided improvement in the British procedure to specify 17 per cent relative humidity, with a tolerance, instead of assuming that trays of saturated $CaCl_2$ will produce and maintain this humidity value.

The authors of the paper have introduced an improvement by defining more closely the end point conditions of the Rapid test and continued to dry the specimens until an equilibrium was attained. However, it might be mentioned that in this respect the National Concrete Masonry Assn. (NCMA) rapid drying method differed from the Am. Concrete Inst. (ACI) rapid drying and the soaking period was longer. Also, the NCMA reference test was based on 50 per cent relative humidity and could properly be called "standard drying" whereas the ACI Reference method tests

TABLE IX.—CONCORDANCE OF TEST RESULTS, AM. CONCRETE INST. REPORT 716 RAPID OVEN-DRYING, HIGH-PRESSURE STEAM-CURED BLOCKS FROM SAME LOT, TESTED AT FIVE LABORATORIES, AVERAGE VALUES.

| Laboratory | Rotary Kiln Aggregate | Cinder Aggregate |
|---|---|---|
| Bureau of Reclamation | 0.042 | 0.038 |
| Bureau of Standards | 0.043 | 0.039 |
| Research Foundation | 0.040 | 0.035 |
| Portland Cement Assn. | 0.043 | 0.038 |
| Lehigh Portland Cement | 0.044[a] | 0.036[a] |

Data from Tables 1 and 2 of *ACI Report 716*, *Journal*, Am. Concrete Inst., Apr., 1953.

[a] Data from Table VIII.

were made at an assumed 10 per cent relative humidity. Although British drying conditions were intended to be the same in both programs the reported shrinkages were based on different criteria of equilibrium length.

TABLE X.—CONCORDANCE OF SHRINKAGE RESULTS PER CENT, USING THREE TEST PROCEDURES; HIGH-PRESSURE STEAM-CURED BLOCK TESTED BY FOUR (OR THREE) PARTICIPATING LABORATORIES, AVERAGE DEVIATIONS EXPRESSED AS PERCENTAGE POINTS SHRINKAGE AND AS PER CENT OF AVERAGE SHRINKAGE.

| Block Producer | Reference Method, 73 F, Saturated NaOH | | British Method, 122 F, Saturated $CaCl_2$ | | Rapid Method, 220 F Oven | |
|---|---|---|---|---|---|---|
| **EXPANDED CLAY AGGREGATE BLOCK** | | | | | | |
| No. 1 | 0.0007 | 3.7 | 0.0014 | 6.7 | 0.0012 | 2.8 |
| No. 3 | 0.0036 | 18.3 | 0.0016 | 7.2 | 0.0017 | 4.7 |
| No. 4 | 0.0024 | 10.5 | 0.0014 | 6.2 | 0.0021 | 5.6 |
| Mean average deviation | 0.0022 | 10.8 | 0.0015 | 6.7 | 0.0017 | 4.4 |
| Shrinkage level, per cent | 0.020 | | 0.022 | | 0.039 | |
| **CINDER AGGREGATE BLOCK** | | | | | | |
| No. 1 | 0.0010 | 5.5 | 0.0028 | 14.0[b] | 0.0012 | 3.2 |
| No. 3 | 0.0056[a] | 27.0 | 0.0026[a] | 10.7 | 0.0026 | 7.0 |
| No. 4 | 0.0069 | 22.7 | 0.0023 | 7.2 | 0.0052 | 10.3 |
| Mean average deviation | 0.0045 | 18.4 | 0.0026 | 10.6 | 0.0030 | 6.8 |
| Shrinkage level, per cent | 0.023 | | 0.025 | | 0.042 | |
| **SAND AND GRAVEL AGGREGATE BLOCK** | | | | | | |
| No. 1 | 0.0017 | 13.4 | 0.0024 | 15.5[b] | 0.0002 | 0.6 |
| No. 3 | 0.0034[a] | 24.6 | 0.0010[a] | 6.5 | 0.0011[a] | 4.9 |
| No. 4 | 0.0026 | 17.6 | 0.0009 | 6.0 | 0.0013 | 7.4 |
| Mean average deviation | 0.0026 | 18.5 | 0.0014 | 9.3 | 0.0009 | 4.3 |
| Shrinkage level, per cent | 0.014 | | 0.015 | | 0.024 | |

Based on the data of Tables 1, 2, and 3 of *ACI Report 716*, *Journal*, Am. Concrete Inst., April, 1953.

[a] Signifies only 3 laboratories reporting data.

[b] The magnitude of these percentages is noteworthy.

The authors state that shrinkage tests with cut prisms were inconsistent and showed poor correlation with whole block. This directs attention to the type of specimen used and possible directional effects. It is assumed that the slender 1 by 1 in. cross-section prisms were cut vertically, but this may be wrong. If this is true, the gage lines on whole block do not represent the same conditions of test and it should not be discouraging if the correlation was not consistent. Some tests have been made in our laboratory to study the correlation of shrinkage of whole block, *horizontal* sections cut from 4-in. size whole block, and *horizontal* prisms 2½ in. wide and face shell thickness cut from whole block.

The results of three successive oven-drying cycles on three small specimens are given in Table XI. It is of interest that the second cycle, under part 2, shows a good check on the first test when compared on the 24-hr soaking basis. Similarly, the third cycle (when measurements were not made after a

24 hr soak) compare favorably with the second cycle. The dry length readings, obtained under closely controlled temperature conditions, show that the final value was about the same after each cycle. Since these units reached the same length each time it seems that they were not subject to progressive change due to repeated oven drying, and this suggests there were no extraneous factors incidental to the testing procedure. The average shrinkages for top section *T*, middle, and bottom section *B* may be compared to determine the top-to-bottom uniformity of the whole block.

TABLE XI.—EXPLORATORY TESTS OF $\frac{1}{3}$ PORTIONS OF 4 BY 8 BY 16-IN. BLOCK CUT HORIZONTALLY, MASONRY UNITS CONTAINING EXPANDED SLAG AGGREGATE.

| Specimen | Total Shrinkage, per cent | | | | | |
|---|---|---|---|---|---|---|
| | 4-in. Section *B* | | 4-in. Section *M* | | 4-in. Section *T* | |
| Gage Line | 1 | 2 | 3 | 4 | 5 | 6 |
| PART 1—FIRST RAPID OVEN TEST SOAKED 24 HR, DRIED 3 DAYS AT 220 F | | | | | | |
| (1) | 0.032 | 0.033 | 0.035 | 0.038 | 0.031 | 0.034 |
| PART 2—SECOND RAPID OVEN TEST, TWO BASES OF SATURATION, DRIED 2 DAYS AT 216 F | | | | | | |
| 24 hr soak | 0.031 | 0.032 | 0.034 | 0.036 | 0.033 | 0.038 |
| (2) 48 hr soak | 0.027 | 0.027 | 0.030 | 0.034 | 0.030 | 0.031 |
| PART 3—BRITISH TEST AND SUBSEQUENT (THIRD) INDIRECT RAPID OVEN TEST, BASIS OF 48 HR SATURATION | | | | | | |
| (3) Final British | 0.012 | 0.016 | 0.014 | 0.014 | 0.012 | 0.014 |
| (4) Indirect rapid | 0.026 | 0.029 | 0.029 | 0.033 | 0.026 | 0.029 |
| DRY LENGTH READINGS OF GAGE LINES AFTER EACH DRYING AS EXPLAINED ABOVE | | | | | | |
| (1) | 0.0766 | 0.0862 | 0.0749 | 0.0675 | 0.0749 | 0.0726 |
| (2) | 0.0772 | 0.0858 | 0.0748 | 0.0677 | 0.0751 | 0.0728 |
| (4) | 0.0774 | 0.0856 | 0.0749 | 0.0677 | 0.0753 | 0.0730 |
| AFTER 48 HR ADDITIONAL DRYING AT 216 F | | | | | | |
| | 0.0773 | 0.0855 | 0.0749 | 0.0677 | 0.0753 | 0.0730 |

The concluding illustration, Table XII, contains data for individual gage lines on various horizontal specimens representing one lot of 2 sizes of block brought to the laboratory at the same time. Length changes in "standard" drying and "oven" drying are included. This tabular data contains enough detail to show that stable length was reached in standard drying. The following brief statements apply to Table XII:

1. Soaking expansion of small specimens was less because these units were wetted at the time of sawing.

2. The data of the two whole block show a slight difference in the oven drying and it would be more specific to compare the cut specimens with the 4-in. block only.

3. The agreement of four gage lines on each whole block assure that the

sections and prisms should be representative.

4. Considering that the sawed specimens were derived from companion block, the agreement of prisms with the 4 in. whole block is satisfactory.

5. It will be noted that the middle portion of units 3 and 5, denoted by letters *M* in the second column, showed slightly higher oven drying shrinkage than the adjacent specimens of same units, there being no indication that measuring shrinkage of prisms of face shell thickness or of $\frac{1}{3}$ portions had any effect on this relationship.

TABLE XII.—SHRINKAGE OF WHOLE BLOCK COMPARED TO SAWED SECTIONS AND PRISMS, SMALL SPECIMENS SAWED WET FROM 4-IN. SIZE UNITS, MASONRY UNITS CONTAINING EXPANDED SLAG AGGREGATE.

Total length changes in per cent.

| Test Unit | Gage Line | Expansion in Soaking | | Dried at 73 F, 50 per cent Relative Humidity | | | | Dried at 216 F | |
|---|---|---|---|---|---|---|---|---|---|
| | | 24 hr | 48 hr | 21 days | 28 days | 35 days | 42 days | 4 days | 7 days |
| 8-in. block | 1t | 0.018 | 0.018 | 0.011 | 0.012 | 0.012 | 0.012 | 0.052 | 0.055 |
| | 2t | 0.022 | 0.022 | 0.016 | 0.016 | 0.016 | 0.016 | 0.055 | 0.059 |
| | 1b | 0.017 | 0.017 | 0.010 | 0.011 | 0.011 | 0.011 | 0.049 | 0.052 |
| | 2b | 0.022 | 0.022 | 0.016 | 0.016 | 0.017 | 0.017 | 0.055 | 0.059 |
| 4-in. block | 1t | 0.014 | 0.015 | 0.009 | 0.010 | 0.010 | 0.010 | 0.045 | 0.050 |
| | 2t | 0.020 | 0.021 | 0.010 | 0.011 | 0.011 | 0.011 | 0.046 | 0.051 |
| | 1b | 0.015 | 0.016 | 0.009 | 0.010 | 0.010 | 0.010 | 0.046 | 0.050 |
| | 2b | 0.016 | 0.017 | 0.008 | 0.009 | 0.009 | 0.009 | 0.042 | 0.048 |
| Prism | 3M 1 | 0.005 | 0.005 | 0.011 | 0.011 | 0.012 | 0.012 | 0.044 | 0.049 |
| | 3M 2 | 0.005 | 0.005 | 0.011 | 0.011 | 0.012 | 0.012 | 0.042 | 0.046 |
| Section | 3B 1 | 0.006 | 0.006 | 0.011 | 0.011 | 0.011 | 0.011 | 0.041 | 0.042 |
| | 3B 2 | 0.005 | 0.005 | 0.012 | 0.011 | 0.012 | 0.012 | 0.038 | 0.040 |
| | 5M 1 | 0.006 | 0.002 | 0.012 | 0.012 | 0.012 | 0.012 | 0.046 | 0.051 |
| | 5M 2 | 0.006 | 0.002 | 0.011 | 0.011 | 0.011 | 0.011 | 0.042 | 0.047 |
| Prism | 5B 1 | 0.005 | 0.004 | 0.012 | 0.012 | 0.012 | 0.012 | 0.041 | 0.044 |
| | 5B 2 | 0.006 | 0.004 | 0.010 | 0.010 | 0.010 | 0.010 | 0.038 | 0.040 |

The shrinkage results with these horizontal type prisms justify further efforts to evaluate their acceptability in future test programs. Other investigators have reported success in using prisms of face shell thickness and, as the authors have stated "the idea of tests on small specimens is attractive from the standpoint of ease in handling, as well as simplification of equipment needs."

MESSRS. E. L. SAXER AND H. T. TOENNIES (*author's closure*).—Mr. Helms has presented additional data on shrinkage measurements which suggest: (1) that the possibility of using prism sections in lieu of whole block for shrinkage specimens should not be abandoned, and (2) that the difference between shrinkage measurements obtained by drying at 216 F and at lower temperatures is simply one of degree and not cause for alarm.

We agree with Mr. Helms on the possibility of cut prism sections in lieu of whole block. Our statement "The use of cut prisms in lieu of whole block did not give uniform results; nor did the

TABLE XIII—COMPARISON OF REFERENCE, RESIDUAL AND INDIRECT RAPID SHRINKAGES.

| Test | Reference Shrinkage, per cent | Residual Shrinkage, per cent | Total (Indirect Rapid) |
|---|---|---|---|
| 1 | 0.044 | 0.036 | 0.080 |
| 2 | 0.032 | 0.036 | 0.068 |
| 3 | 0.048 | 0.039 | 0.087 |
| 4 | 0.024 | 0.029 | 0.053 |
| 5 | 0.037 | 0.038 | 0.075 |
| 6 | 0.018 | 0.032 | 0.050 |
| 7 | 0.035 | 0.038 | 0.073 |
| 8 | 0.017 | 0.032 | 0.049 |
| 9 | 0.032 | 0.033 | 0.065 |
| 10 | 0.024 | 0.033 | 0.057 |
| 11 | 0.042 | 0.040 | 0.082 |
| 12 | 0.024 | 0.037 | 0.061 |
| 13 | 0.047 | 0.034 | 0.081 |
| 14 | 0.028 | 0.035 | 0.063 |
| 15 | 0.022 | 0.030 | 0.052 |
| 16 | 0.017 | 0.025 | 0.042 |
| 17 | 0.026 | 0.035 | 0.061 |
| 18 | 0.022 | 0.028 | 0.050 |

average shrinkage values thus obtained show a consistent relationship to those from whole block" applied only to the prisms used in our tests. The statement was not intended to apply to all shapes and sizes of cut sections possible from a concrete block. Future research of shrinkage test methods now in plan by NCMA include further study of cut sections.

We do not agree with Mr. Helms' conclusion that oven temperature effect is a matter of degree. Our data indicate that the shrinkage which accompanies oven drying (220 F) of block already dried to an equilibrium condition with air at 50 per cent relative humidity and 73 F (Reference test) has little or no relationship to the shrinkage obtained by Reference drying. This can be seen in Table IV of our report. The residual shrinkage of autoclaved and low-pressure steam-cured block are nearly the same for any particular aggregate listed in the table. However, the shrinkage with Reference drying block of these two curing methods is very different (see Table I). It is the adding of these "nearly the same residual shrinkages" to the Reference values which accounts for the increase from 0.47 to 0.69 in the average HP/LP ratio of Table III.

We have obtained the residual shrinkage of many specimens in recent months in the course of another research program. The results of eighteen of these tests are listed in the accompanying Table XIII below. They indicate clearly that the residual shrinkage which accompanies oven drying has no relationship to that accompanying normal Reference drying. The fact that total (indirect rapid) shrinkages correlate with Reference shrinkages lies in the Reference shrinkages themselves despite the "sameness" of residual values. To judge high- and low-shrinkage block by the indirect rapid values would not be as sensitive a method as it would by the Reference values.

# PERFORMANCE TESTS OF CONCRETE TRUCK MIXERS*

By Albert G. Timms[1]

## Synopsis

This report describes the performance of concrete truck mixers of the inclined-axis type when tested under field conditions. The effects of nine different batching sequences on the uniformity of both air-entrained and non-air-entrained concrete were investigated. These were split loading, ribbon loading, and combinations of these two. In some batches water was added at the batching plant and in others it was added from the water tank of the truck mixer at the job site. Tests were made with mixers loaded to their 4½-cu yd normal rated capacity and parallel tests were made for loads of 5 cu yd, which these mixers are guaranteed to handle. The number of revolutions of the mixer drum was varied in steps from 50 to 150. The times required for loading, discharging and mixing were all measured. The concrete was sampled from the discharge chute at the start of discharge, at the mid-point and near the end of discharge. The three samples of concrete from each batch were tested for slump, unit weight, air content, and strength; wash tests were made to determine uniformity of proportions and grading changes of the aggregates. The tables and figures contain data which show the influence upon the resulting concrete of methods of mixer loading in combination with the other variables.

The results of the tests clearly demonstrated that uniform pavement-type concrete with slumps below 2 in. can be made and discharged reasonably rapidly without significant segregation of the ingredients. For satisfactory uniformity, the minimum amount of revolutions of the drum recommended by the ASTM specifications[2] appeared sufficient for the majority of the loading methods; improvement in uniformity was obtained by increasing the number of revolutions of the mixer drum. The tests indicated the importance of having water in the drum for both ribbon and split loading.

Truck mixers have been in general use in the United States for over 30 years, and their use has been continually expanding. The present operating standards for truck mixers are based on data collected when the industry was young and on operating experiences over the years. The only recent comprehensive investigation previous to the present series was based on tests of eleven truck mixers reported by Stanton Walker and D. L. Bloem (**3**).[3] Lack of understanding by some operators of the requirements

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Consulting Engineer, Worthington Corp., Plainfield Division, Plainfield, N. J.

[2] Tentative Specifications for Ready-Mixed Concrete (C 94 – 55 T), 1955 Book of ASTM Standards, Part 3, p. 1269.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1026.

TABLE I.—PROPERTIES OF CEMENT AND AGGREGATES.

Cement

Average of mill test reports from eleven carload shipments.

Physical

| | Specific Surface | Setting Time | | Autoclave Expansion, per cent | Compressive Strength, 1-2.75 Mortar, psi | | Tensile Strength, 1-3 Mortar, psi | |
|---|---|---|---|---|---|---|---|---|
| | | Initial Hr: min | Final Hr: min | | 3 days | 7 days | 3 days | 7 days |
| Average | 3041 | 2:25 | 4:30 | 0.227 | 2638 | 3674 | 354 | 425 |
| Range | 173 | 0:30 | 0:30 | 0.138 | 666 | 1133 | 75 | 56 |
| Average Range, per cent | 6 | 20 | 11 | 61 | 25 | 31 | 21 | 13 |

Chemical

| | Chemical Analyses, per cent | | | | | Loss on Ignition, per cent | Insoluble Residue, per cent | Compound Composition, per cent | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Fe_2O$ | MgO | $SO_2$ | | | $C_3S$ | $C_2S$ | $C_3A$ |
| Average | 19.80 | 6.93 | 3.14 | 2.60 | 2.53 | 0.74 | 0.20 | 45.21 | 22.76 | 13.04 |
| Range | 0.46 | 1.12 | 0.45 | 0 | 0.41 | 0.56 | 0.02 | 7.56 | 4.90 | 2.52 |
| Average Range per cent | 2 | 16 | 14 | 0 | 16 | 75 | 10 | 17 | 22 | 19 |

Aggregates

Sieve analyses are averages of 75 tests made throughout the progress of the investigation. Specific gravities and absorptions are averages of tests made at the beginning of the investigation.

| Item | 3/4-in. Stone | Sand |
|---|---|---|
| Grading, per cent passing: | | |
| 1 in. | 100 | ... |
| ¾ in. | 87 | ... |
| ½ in. | 19 | ... |
| ⅜ in. | 6 | ... |
| No. 4 | 0 | 100 |
| No. 8 | ... | 96 |
| No. 16 | ... | 83 |
| No. 30 | ... | 54 |
| No. 50 | ... | 12 |
| No. 100 | ... | 2 |
| Fineness Modulus | 7.08 | 2.53 |
| Specific Gravity, bulk SSD | 2.88 | 2.62 |
| Absorption, per cent | 1.0 | 1.1 |

for adequate mixing has raised the question by consumers of the ability of the truck mixer to produce concrete of low slump consistency.

The present report describes the completed portion of an investigation of the performance of concrete truck mixers of the inclined-axis type tested under field conditions. Two phases of the ready-mixed concrete problem have been studied—the method of loading which would appear to be best to expedite mixing and the number of revolutions required to do an adequate mixing job. Low slump concrete suitable for paving operations was used in the tests. The concrete left over after samples had been taken was used to pave the storage yard of the Plainfield, N. J., plant of the Worthington Corp.

## SCOPE OF INVESTIGATION

Two basic concrete mixes were used—one with entrained air and the other without. Nine different methods of loading were used consisting of ribbon and split loading and combinations of these which would represent the conditions encountered in one and two stop ready-mix loading plants. Ribbon loading is the adding of all or certain combinations of materials in a single stream, with the rate of flow for each adjusted so they enter the mixer simultaneously. Split loading is the adding of the materials separately. Two batches of concrete were mixed for each test condition. In the major project three rates of mixing were used, 4, 7, and 10 rpm. Only the 10 rpm tests have been reported in this paper. The time of mixing was varied between 50 and 150 revolutions. In the discussion of the results, each loading method is described separately.

Slump, unit weight, strength, and washout determinations were made, and where air entrainment was involved air determinations were made. Charging and discharge times were noted as well as temperatures of the concrete and air temperatures and relative humidities.

## EQUIPMENT AND TESTING FACILITIES

Most of the tests were made with truck mixers of $4\frac{1}{2}$-cu yd rated capacity which were loaded through the end. The mixers were operated at a speed of 10 rpm. Representative tests were made with five inclined-axis truck mixers, each of a different make.

A full-scale batch plant was constructed to produce either ribbon loading or split loading. The aggregate plant contained three 24-ton compartments with three scale arms for successive weighing of the ingredients in a 3-cu yd weighing hopper.

A moist room approximately 10 by 20 ft was constructed in an insulated warehouse for curing the 6 by 12-in. concrete test specimens. The room was equipped with atomizer sprays and temperature control. Equipment was available for wet sieving and for making slump, unit weight, and air determinations of the concrete. Heavy well-waxed paper molds were used for making the 6 by 12-in. concrete test specimens. One day after molding, the specimens were capped on both ends with neat cement using plate glass to give plane ends. A hydraulic testing machine equipped with a spherical bearing block was used for the compression tests.

## CONCRETE MATERIALS

The cement used was a type 1 portland cement purchased in carload lots. The average results of the mill tests of the cement are given in Table I. The sand was from a local pit and quite uniform in grading. The coarse aggregate was crushed trap rock. The average grading and specific gravity of the aggregates are also shown in Table I.

## CONCRETE MIXTURES

The base mix was designed to give a cement content of 6 sacks per cu yd. For each batch the moisture in the sand was determined and the batch weights corrected to take care of the water in the sand. No correction was made for water carried by the coarse aggregate. An electric moisture meter was installed in the sand weighing hopper that indicated the moisture content directly. The meter was checked against a weighed sample daily. The capacity of the batcher was such that, for most mixes, it was necessary to load the aggregates in two portions. Measurements of moisture on the first portion of the batch permitted adjustments in aggregate quantities to be made on the second portion, thus providing the desired quantity of aggregates exclusive of free water.

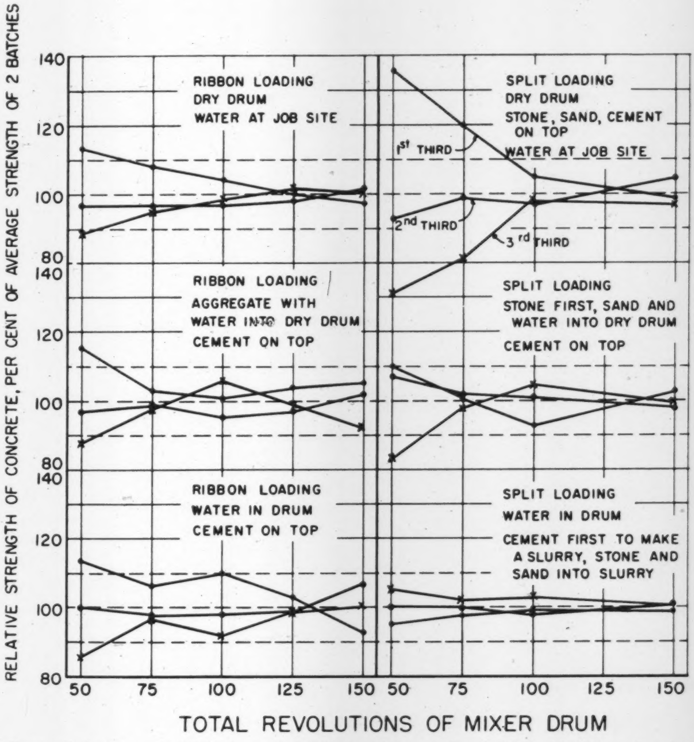

FIG. 1.—Effect of Amount of Mixing on Relative Strength of Non-Air-Entrained Concrete with $4\frac{1}{2}$-cu yd Load for Different Batching Conditions.

When air entrainment was used, the sand content of the base mix was reduced to compensate for the volume of air and lower water content needed for the same degree of consistency and still maintain the cement content of 6 sacks per cu yd. Since the maximum size of the coarse aggregate was $\frac{3}{4}$ in. (87 per cent passing the $\frac{3}{4}$-in. sieve), an attempt was made to keep the air content near the 6 per cent limit usually specified for pavement concrete.

### SAMPLING THE CONCRETE

The concrete was sampled during the discharge of the concrete from the mixer onto the subgrade by passing a shovel at the end of the chute through the entire stream of concrete. Samples were taken during the first third of the load, the second third, and the last third and each placed in a separate mixing pan large enough to hold a 300-lb sample. The sampling procedure was in accordance with the ASTM Standard Method of Sampling Fresh Concrete (C 72 – 54).[4]

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[4] 1955 Book of ASTM Standards, Part 3, p. 1368.

The concrete was remixed with a shovel just enough to correct any segregation due to the sampling. Immediately after remixing, a slump test was made. The remainder of the sample was used to make a unit weight test, to cast three concrete cylinders for a strength test, and finnally a wash test.

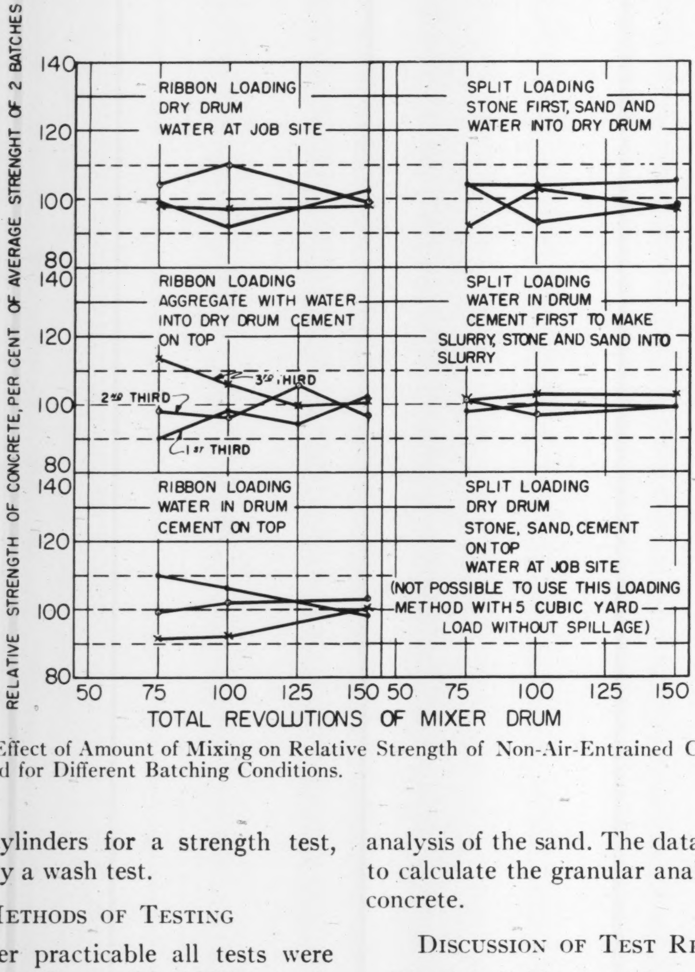

Fig. 2.—Effect of Amount of Mixing on Relative Strength of Non-Air-Entrained Concrete with 5-cu yd Load for Different Batching Conditions.

## Methods of Testing

Whenever practicable all tests were made in accordance with ASTM standard methods.

The wash test consisted of washing a sample of concrete over a reinforced No. 100 sieve topped by a No. 4 sieve. The concrete sample was weighed before washing, and the stone portion and sand portion weighed separately after washing. The materials were dried overnight and sieve analyses made of the stone and sand samples. The entire stone sample was used in the sieve analysis, and a 500-g sample, previously dried over a hot plate, was used for the sieve analysis of the sand. The data were used to calculate the granular analysis of the concrete.

## Discussion of Test Results

The data in Figs. 1 to 9 are a summary of a large number of tests. Summary tables and tables of the individual results are available in mimeograph form.

Testing a truck mixer load of concrete is a time-consuming project, and for this reason the range in variables had to be restricted to the number that could be tested in a single construction season. Only two batches were made for each condition because of the restriction of time, even though it is known that under well-controlled laboratory tests the minimum should not be fewer than three.

Studies of the uniformity of the strength are based on ratios of strength for each given third as compared with the average strength for the batch. This procedure was indicated by the inevitable variations in strength level for batches made at different times and under widely varying conditions. The tests were conducted over the period of June to December, and there were significant variations in weather. Eleven different carloads of cement were used, and the strength-producing properties of the different shipments varied substantially (see Table I for range in mill tests). The variations in strength within a given third were, in general, small as compared with the variations between thirds.

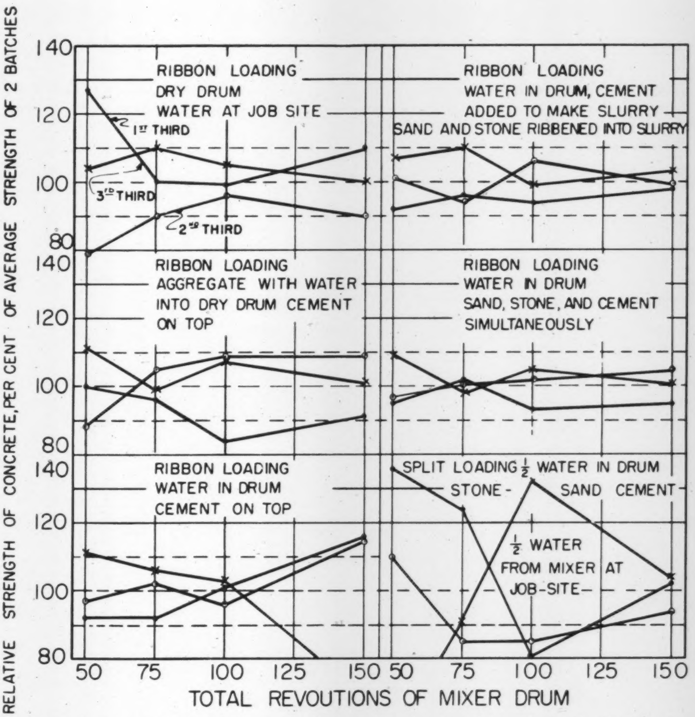

FIG. 3.—Effect of Amount of Mixing on Relative Strength of Air-Entrained Concrete with 4½-cu yd Load for Different Batching Conditions.

There is no generally accepted standard as to the most significant measure of mixing efficiency. Walker and Bloem used aggregate of 1½-in. maximum size and chose the range in plus ¾-in. aggregate (the material retained on a ¾-in. sieve) from the "washout" test as the best single measure of over-all variation of within-batch composition. Since the maximum size of aggregate in this investigation was ¾ in., it was decided to use the plus No. 4 material as a measure of within-batch composition. The No. 4 material (that which was retained on a No. 4 sieve) represents the percentage of stone in the concrete. The range in strength,[5] the coefficient of variation of the strength,[6] and the grading factor were also studied in detail. The grading factor was computed from the sieve analysis of the wash-out test in a manner similar to the determination of the fineness modulus (see Figs. 7 and 8).

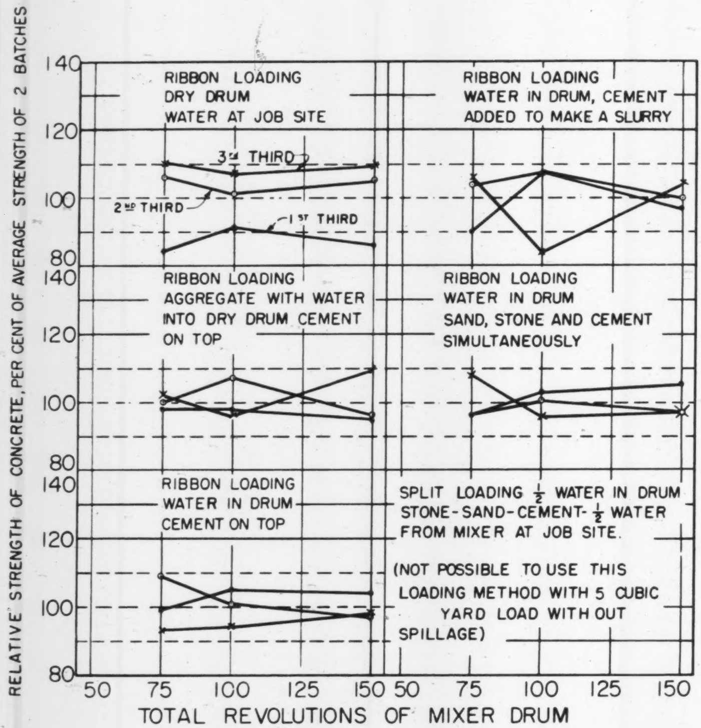

FIG. 4.—Effect of Amount of Mixing on Relative Strength of Air-Entrained Concrete with 5-cu yd Load for Different Batching Conditions.

*Effect of Methods of Loading:*

The effect of methods of loading on relative strength and consistency of concrete and grading of the aggregate after washing out the cement are shown in Table II and Figs. 1 to 8. The data represent 86 batches of non-air-entrained concrete and 78 batches of air-entrained concrete mixed under comparable conditions.

It was believed that concrete within the ranges in relative strength of ±10 per cent would indicate the area of uniform concrete. To assist in making a study of the diagrams, these 10 per cent lines were added.

The coefficient of variation of the within-batch uniformity of the strength

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[5] The range in compressive strength is the difference between the maximum and minimum average strengths of the thirds, expressed as a percentage of the over-all strength of the entire batch.

[6] Coefficients of variation are calculated from the formula,

$$c = \frac{\sqrt{\frac{\Sigma(\bar{x} - x)^2}{n}}\;100}{\bar{x}}$$

where $\bar{x}$ is the average strength for the entire batch, $x$ is the average strength for a given third and $n$ is equal to 3.

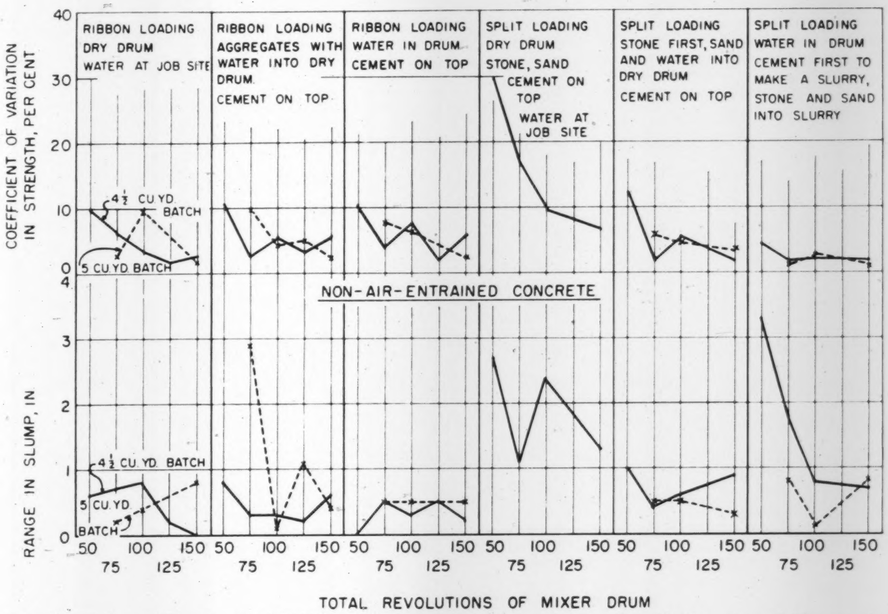

FIG. 5.—Effect of Amount of Mixing and Methods of Loading on the Uniformity of Slump of the Concrete and Coefficient of Variation in Strength for Non-Air-Entrained Concrete.

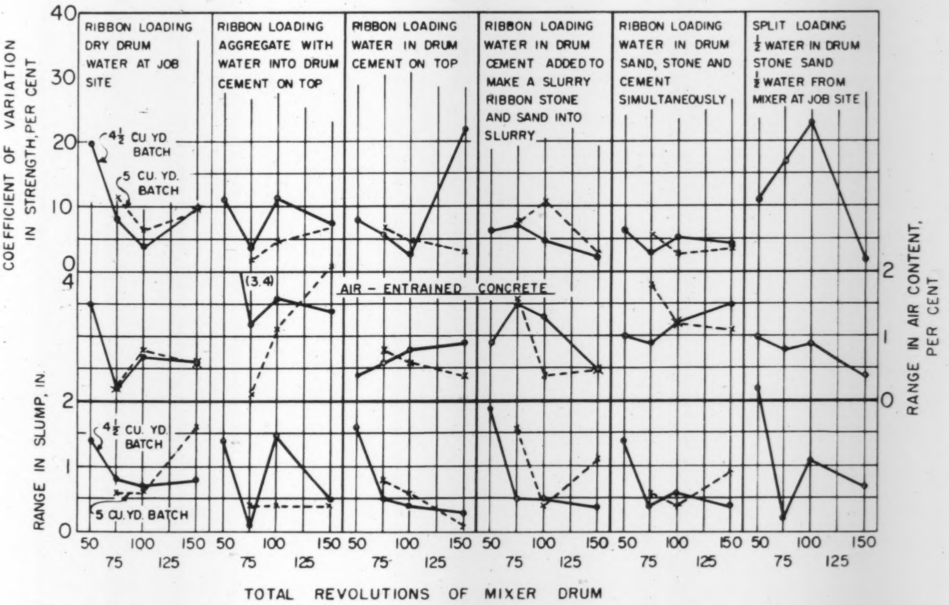

FIG. 6.—Effect of Amount of Mixing and Methods of Loading on the Uniformity of Slump of the Concrete, Air Content and Coefficient of Variation in Strength.

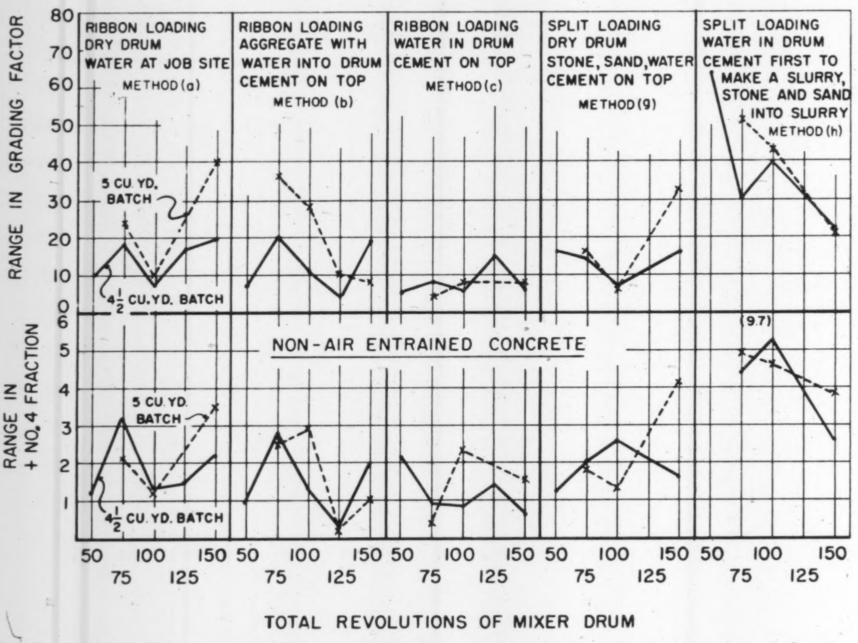

FIG. 7.—Effect of Amount of Mixing and Methods of Loading on the Uniformity of the Grading of the Ingredients in Non-Air-Entrained Concrete.

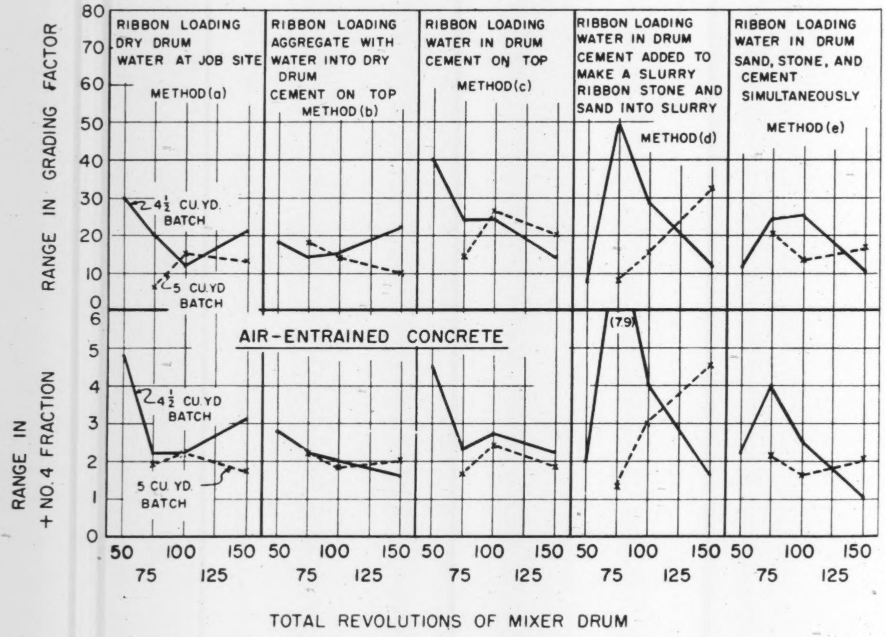

FIG. 8. Effect of Amount of Mixing and Methods of Loading on the Uniformity of the Grading of the Ingredients in Air-Entrained Concrete.

was also calculated but was not of much value in assessing the influence of method of loading. The coefficients of variation are shown in Figs. 5 and 6 and confirm the conclusions drawn from the relative strengths.

As pointed out earlier, the tests were designed so that the average slump would be less than 2 in.; consequently differences in slump were small. The range in slump was considered to be a measure of the uniformity of the consistency within the batch.

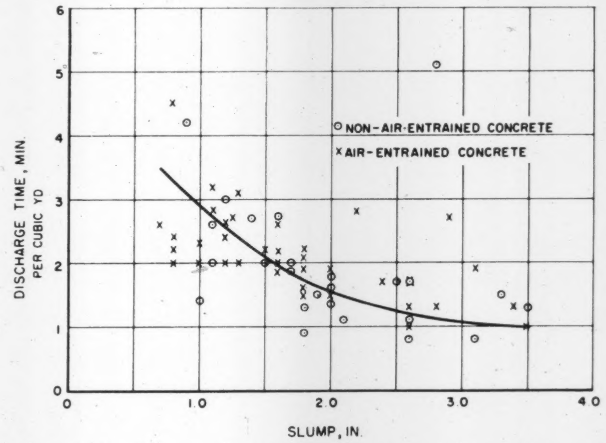

FIG. 9.—Relation Between Consistency of Concrete and Discharge Time.

Segregation of the aggregate was indicated by the range in the grading factor. In the author's opinion, a range of more than 20 is indicative of segregation within the batch. The range in plus No. 4 fraction also was used as a measure of segregation with more than five percentage points considered to indicate objectionable segregation.

The results for each of the nine loading methods follow.

## RIBBON LOADING

*Method (a)—Non-air- and air-entrained concrete:*

Starting with a dry drum, the stone, sand, and cement were ribboned into the mixer and the water was added from the mixer tank at the job site. As may be seen from Figs. 1 and 2, all the non-air-entrained concrete mixed 75 revolutions or longer was within the ±10 per cent range indicating satisfactory uniformity. The 4½-cu yd loads of air-entrained concrete (Fig. 3) mixed 75 revolutions or more were also within the ±10 per cent. The 50 revolutions of the mixer drum were not enough to obtain the arbitrary degree of uniformity used in these studies. For the air-entrained concrete, the 5-cu yd batches (Fig. 4) were slightly outside the ±10 per cent range.

The within batch consistency of the concrete (Figs. 5 and 6) for this method of loading varied little as indicated by the range in slump—being less than 1 in.—except for the air-entrained concrete, where in two cases it was between 1 and 2 in.

In general, the range in grading factor (Figs. 7 and 8) was below 24, with most of the values within the proposed limit of 20. The two exceptions were 40 for the 5-cu yd load and 150 revolutions with non-air-entrained concrete and 30 for the 4½-cu yd load and 50 revolutions of the air-entrained concrete. In no case did the plus No. 4 material exceed 5 percentage points.

*Method (b)—Non-air- and air-entrained concrete:*

Starting with a dry drum, the stone, sand, and water were ribboned into the mixer with the cement added on top. For the 4½-cu yd batch, the results were similar to those obtained with loading method (*a*) in that the concrete mixed 75 revolutions or longer was usually within ±10 per cent in range for relative strength, while 50 revolutions did not quite give the desired uniformity. There were three exceptions in the case of the air-entrained concrete where the values were outside the ±10 per cent range (see Fig. 3).

TABLE II.—COMPARISON OF DIFFERENT INCLINED-AXIS TRUCK MIXERS.
In general, all results are for one test only.

| Mixer | Mixer Speed, rpm | Total Revolutions—after Water | Range in Grading Factor | Range in +(No. 4), per cent | Range in Slump, in. | Relative Strengths, per cent | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | 1st Third | 2nd Third | 3rd Third |
| METHOD (*b*) RIBBON LOADING-DRY DRUM-STONE, SAND AND WATER-CEMENT ON TOP (4½ CU YD) | | | | | | | | |
| A | 12 | 75 | 11 | 0.8 | 0.6 | 96 | 110 | 96 |
| B[a] | 10 | 75 | 7 | 3.2 | 1.0 | 101 | 103 | 96 |
| C | 12 | 75 | 17 | 1.7 | 0.2 | 104 | 92 | 104 |
| D | 10 | 75 | 28 | 3.0 | 1.0 | 97 | 95 | 108 |
| E | 10 | 75 | 20 | 2.8 | 0.3 | 103 | 99 | 98 |
| METHOD (*g*) SPLIT LOADING-DRY DRUM-STONE, SAND AND WATER-CEMENT ON TOP (5 CU YD) | | | | | | | | |
| A | 10 | 100 | 66 | 10.7 | 0.8 | 119 | 90 | 91 |
| B | 10 | 100 | 91 | 13.4 | 1.8 | 112 | 113 | 75 |
| C | 10 | 100 | 17 | 1.0 | 0.3 | 110 | 99 | 92 |
| D | 10 | 100 | 27 | 2.3 | 1.5 | 156 | 89 | 54 |
| E | 10 | 100 | 6 | 1.3 | 0.5 | 104 | 93 | 103 |
| METHOD (*f*) SPLIT LOADING-DRY DRUM-STONE-SAND-CEMENT ON TOP-WATER FROM MIXER TANK AT JOB SITE (5 CU YD) | | | | | | | | |
| A | 10 | 100 | 65 | 10.2 | 8.0 | 72 | 125 | 102 |
| B[b] | 10 | 100 | 75 | 8.5 | 0.1 | 95 | 112 | 93 |
| C | 10 | 100 | 65 | 7.4 | 1.2 | 97 | 104 | 99 |
| D[c] | 10 | 100 | 36 | 5.0 | 0.5 | 116 | 89 | 95 |
| E | 10 | 100 | ... | ... | ... | ... | ... | ... |

[a] This mixer had a rated capacity of 5-cu yd and a ½-cu yd larger batch than indicated was used throughout.

[b] Could not get batch into mixer, 4½-cu yd load used.

[c] Water added at plant in order to move mixer without further loss of cement, also used 4½-cu yd load.

The consistency of the concrete was very uniform (Figs. 5 and 6); most batches showed a range in slump of less than 1 in. The one exception—the 5-cu yd batch of non-air-entrained concrete mixed for 75 revolutions—had a range in slump of 2.9 in.

The range in grading factor (Fig. 7) for the non-air-entrained 4½-cu yd batches of concrete was within the arbitrary value of 20. For the 5-cu yd batch of non-air-entrained concrete, the grading factor exceeded the 20 point limitation for the concrete mixed both 75 and 100 revolutions. The air-entrained concrete (Fig. 8) was within the range of 20 points except for the 4½-cu yd concrete mixed 150 revolutions which had a factor of 22. In no case did the plus No. 4 fraction exceed the 5 percentage points, indicating that the ratio of fine to coarse aggregate remained quite constant.

*Method (c)—Non-air- and air-entrained concrete:*

Starting with water in the mixer drum, the stone and sand was ribboned into the water, with the cement on top. For the non-air-entrained concrete (Figs. 1 and 2), mixing 75 revolutions or longer produced the desired uniformity in relative strength, while 50 revolutions proved insufficient. For air-entrained concrete (Figs. 3 and 4), 75 and 100 revolutions of the mixer drum gave the desired uniformity in strength. The 50 revolutions were not quite enough to meet the ±10 per cent range. However, in the case of the $4\frac{1}{2}$-cu yd load, the 150 revolutions produced poor uniformity as indicated by relative strengths of 116 and 69. Only one of the two duplicate batches showed very poor uniformity. It is probable therefore that the poor average uniformity for 150 revolutions is accidental and does not indicate any significant trend. With the 5-cu yd load, the uniformity in strength was satisfactory for all mixing times.

The range in slump for this method of loading (Figs. 5 and 6) varied from 1.6 to 0.0 in., indicating a high degree of uniformity in the consistency of the concrete.

With this method of loading, only in the air-entrained concrete (Fig. 7) was there any evidence of marked segregation of the aggregate. The worst case was the $4\frac{1}{2}$-cu yd load mixed 50 revolutions which gave a value of 40. The plus No. 4 values, while all below 5 percentage points, also indicated the greatest variation with the batch mixed 50 revolutions.

*Method (d)—Air-entrained concrete only:*

Starting with water in the drum, cement was added to make a slurry. The stone and sand were ribboned into the slurry. For each mixing time, the relative strength uniformity (Figs. 3 and 4) was within the desired limits, except for the 5-cu yd batch mixed 100 revolutions.

The consistency of the concrete was quite uniform (Fig. 6) as indicated by the range in slump from 1.9 to 0.4 in. with the majority of the values less than 1 in.

Considerable variation in the grading (Fig. 8) was found for this method of loading. This was reflected in both the grading factor and the plus No. 4 material. At first glance this does not appear to be compatible with the high degree of uniformity found by the strength tests. Since water-cement ratio determines the strength, it would be reasonable to suppose that in a slurry mix, this would be uniform but that the amount of paste in the concrete could vary considerably without affecting the strength.

*Method (e)—Air-entrained concrete only:*

Starting with water in the drum, the stone, sand, and cement were added simultaneously. For all conditions studied, the relative strengths (Figs. 3 and 4) were within the limits of uniformity proposed.

The consistency as measured by the slump test was very uniform (Fig. 6), with the slump ranging from 1.4 to 0.4 in.

For this method of loading the grading (Fig. 8) remained quite uniform, for only two of the conditions were the factors over 20 and these were 24 and 25. The plus No. 4 material was within the 5 percentage point limit. In general, the grading factors tend to be a little higher for air-entrained concretes than for non-air-entrained.

From the point of view of over-all uniformity, this probably is the most desirable method of loading a mixer.

### SPLIT LOADING

*Method (f), Non-air-entrained concrete only:*

Starting with a dry drum, the stone was loaded first, then the sand, and the cement on top; the water was added from the mixer tank at the job site. This method of loading is very unsatisfactory because the ingredients do not settle, and when the mixer is moved even the 4½-cu yd batches throw out some cement. This method could not be used with the 5-cu yd load because it was not possible to get all the cement into the mixer. After the water was added, it required at least 100 revolutions of the drum to give the desired uniformity. The relative strength (Fig. 1) for those batches mixed 50 and 75 revolutions was very erratic.

The range in slump (Fig. 5) varied from 2.7 to 1.1 in. The difference in consistency from the start of discharge to the end could be readily detected by visual observation. For the shorter mixing periods the start of discharge was much drier than the last of the load.

The greatest amount of segregation (Fig. 7) as indicated by the range in the grading factor was found in this method of loading; however, the results show this is largely overcome when the mixing is continued to 150 revolutions. The range in plus No. 4 material reflects the same trend as the grading factor.

*Method (g)—Non-air-entrained concrete only:*

In a dry drum, first the stone was loaded, then the sand and water together, with the cement in last on top of the aggregate. The relative strengths of all concrete mixed 75 revolutions or longer were within the ±10 per cent range. The 50 revolutions were not quite enough to produce the uniformity in the relative strengths desired.

The consistency (Fig. 5) was uniform throughout the batches with the slumps ranging from 1.0 to 0.3 in.

The ranges in grading factors (Fig. 7) for this method were all low except for the 5-cu yd batch mixed 150 revolutions. This high value is not believed to be significant of any tendency for long mixing periods to be harmful. The range in the plus No. 4 material reflects the results obtained with the grading factor.

*Method (h)—Non-air-entrained concrete only:*

Water was placed in the drum first and then cement added to make a slurry. First the stone and then the sand was added separately. The relative strength (Fig. 1) of all of the concrete was within the ±10 per cent range regardless of size of batch or number of revolutions of the drum.

In general, the consistency (Fig. 5) was quite uniform except in the case of the batch mixed 50 revolutions where the range in slump was 3.3 in.

The wash-out test indicated extensive segregation of the aggregate. This is shown by the high values for the grading factor (Fig. 7) and plus No. 4 material. This slurry mix was similar in this respect to the slurry mix in method (*d*). It also showed improvement in uniformity of grading with increase in the number of revolutions of the mixer drum.

*Method (i)—Air-entrained concrete only:*

One-half of the required water was placed in the drum. Then in this order, stone, sand, and cement were added separately and the remainder of the water was added from the mixer tank at the job site. It was not possible to use this loading method with 5-cu yd loads without spillage.

The relative strengths were very erratic and it required 150 revolutions of the drum to bring the relative strengths within the ±10 per cent range.

The consistency of the concrete (Fig. 6) varied more than for the other methods of loading. The range in slump was from 2.2 in. for 50 revolutions to 0.2 in. for 75 revolutions.

The range in grading factor (Fig. 8) varied from 63 for 50 revolutions to 10 for 75 revolutions but was up to 34 for the 150 revolution batch. This variation in grading uniformity was paralleled by the range in plus No. 4 material from 7.4 for 50 revolutions to 1.5 for 75 revolutions.

### COMPARISONS OF FIVE DIFFERENT INCLINED-AXIS TRUCK MIXERS

Tests using three methods of loading were carried out on mixers made by five different manufacturers (see Table II). The first group of tests with rated loads only, used method (*b*) ribbon loading of aggregate, then water, and the cement on top. With this method uniform concrete as indicated by the relative strengths and the range in plus No. 4 material was obtained. No difficulty was encountered in batching, and the concrete at discharge also indicated good uniformity. The larger loads were not used with this method because no difficulty would be expected.

The second group employing split loading method (*g*) with 5 cu yd started with a dry drum, stone, sand, and water, with the cement on top. Difficulty was encountered with all mixers in this method of loading. Mixer A threw out about 0.5 per cent of the cement and at discharge the concrete appeared a little dry at the start but was good thereafter. Mixer B threw out about 8 per cent of the cement, the concrete was very dry at the start of discharge, and after the first sampling 10 gal of additional water was added. Mixer C also lost about 8 per cent of the cement, but the concrete was quite uniform as indicated by the tests and by observation. Mixer D threw out a very small amount of cement (less than 0.5 per cent) and the mixing was poor; at the start of discharge the concrete was very rich in cement and at the end lean and stony. This lack of uniformity was clearly brought out in the wide variation in the relative strengths. Mixer E also threw out about 8 per cent of the cement; however the concrete appeared quite well mixed. This was also shown by the uniformity of the relative strengths and the range in plus No. 4 materials.

The third group used split loading method (*f*) with the 5-cu yd load and the water added at the job site. All the mixers threw out excessive amounts of cement (as much as 25 per cent). Only in mixers A and C was it possible to get all the cement in the mixer, let alone move the mixers to the job site without additional spillage. The concrete from mixer A was poor, very wet at the start, and dry and stony at the end. The consistency of the concrete from mixer C also varied from dry to wet. This method of loading was not used in the general investigation because the resulting concrete was so poorly mixed that it was not satisfactory for paving the storage yard at the plant.

### RELATION BETWEEN SLUMP AND DISCHARGE TIME

The ready-mixed concrete operator is much interested in the discharge time of truck mixers as affected by consistency, many operators claiming that the discharge time for low-slump concrete is excessively slow. These tests presented a good opportunity to measure the discharge time for both air-entrained and non-air-entrained concrete. The relationship between slump and discharge time in minutes per cubic yard is shown in Fig. 9. The average curve is quite flat for slumps greater than 2 in. but goes up quite rapidly for slumps less than 2 in.

The time of discharge for a concrete of 1-in. slump is about double that for a 2-in. slump concrete but less than 3 min per cu yd.

### CONCLUSIONS

Tests in which various methods were used in loading truck mixers and where the amount of mixing was varied in steps from 50 to 150 revolutions demonstrated the importance of these two factors in determining the uniformity of the concrete throughout the batch.

The detailed conclusions are as follows:

1. Good uniformity, as measured by variations in relative strength, was obtained when the water was introduced into the mixer at the batching plant. This was true for both non-air-entrained and air-entrained concrete, for ribbon and split loading, for the amounts of mixing investigated, and for 5-cu yd loads as well as $4\frac{1}{2}$-cu yd loads.
2. The different methods of measuring uniformity indicated that the minimum of 50 revolutions of the drum are, except for certain slurry and split loading conditions, sufficient to produce the uniformity desired.
3. Under the poorest of the loading conditions represented by these tests, improvement in uniformity of relative strength was obtained by increasing the number of revolutions of the mixer drum.
4. The uniformity of consistency as measured by the slump tests shows that a paving type concrete of low slump (less than 2 in.) can be produced readily in truck mixers. The relationship between slump and discharge time shows that even low slump concretes can be discharged rapidly without segregation of the ingredients.
5. Lack of uniformity in the grading of the ingredients appeared to occur only when slurry mixes were employed. Increasing the amount of mixing improved the uniformity. Some results showed that for other than the slurry mixes there was a tendency for the 150 revolutions to cause some slight segregation.
6. In this investigation, the mixers performed as well when mixing a 5-cu yd as for $4\frac{1}{2}$-cu yd loads; the one exception was the case of split loading where water was added at the job site.

### ADDITIONAL TESTS

Other factors influencing the performance of truck mixers were studied, most important of which was rate of mixing. The data of these tests will be presented in another paper.

*Acknowledgments:*

The tests described were planned by K. W. Horsman, General Manager, L. G. Hilkemeier, Manager of Engineering, and J. H. Bealer, Assistant Manager of Engineering, Plainfield Division, Worthington Corp.

The work was under the supervision of the author, assisted by Roger Green, who also helped in the preparation of the data. Outstanding work was done by Al Pearse, who acted as general foreman, supervised the batching, and operated the truck mixer.

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### REFERENCES

(1) S. C. Hollister, "Tests of Concrete from A Transit Mixer," *Journal*, Am. Concrete Inst., Feb., 1932, *Proceedings*, Vol. 28, p. 405.

(2) E. O. Sweetser and A. W. Brust, "Report of Performance of a Transport Mixer," Washington University (1938).

(3) Stanton Walker and D. L. Bloem, "Tests of Concrete Truck Mixers," Mimeographed Progress Report of the National Ready-Mixed Concrete Assn., Feb., 1953.

(4) Stanton Walker and D. L. Bloem, "Tests of Concrete Truck Mixers," Compilation of Detail Data, Nat. Ready-Mixed Concrete Assn., Sept. 1953.

# DISCUSSION

Mr. A. D. Conrow.[1]—Was there any particular difficulty in cleaning the mixer, that is, completely discharging the batch with a stiff mix?

Mr. Albert G. Timms (*author*).—We made only one test, which we called a holdback test, to determine the amount of material retained in the drum. We found that it took so much time and required so much water that it was not feasible. We experienced no difficulty in flushing out the mixer after each batch. This was true for all the mixers tested. They all seemed to discharge the stiff mix almost as well as they did the wetter ones. However, the time of discharge was somewhat slower.

Mr. John J. White.[2]—In washing out the drum, what assurance have we that a considerable percentage of water still does not remain in the drum, and what are the methods for insuring a dry drum?

Mr. Timms.—I do not know that I am qualified to answer that question. In our tests we let the drum run in the discharge direction long enough so that when we moved the mixer, we could not hear any water swishing around in it. For all practical purposes, we had a dry drum.

Mr. Glenway Maxon[3] (*by letter*).—The customary method of sampling from the first third, second third, and last third permit of unrealistic division where the amount of deviation from the average is being sought. In this respect, the 1955 Concrete Manual of the Bureau of Reclamation states, "The samples are taken from the front and back of the concrete surface with the mixer stopped, or they may be taken from the first and last portions of the batch as discharged from the mixer. The method depends on the type of mixer and its setting." The assumption made herein is that the interior deviates if the extremities are at variance, and where the extremities are uniform, the interior will be uniform, an assumption which has been borne out pretty well from numerous tests. If it is left to judgment as to finding the exact center of the thirds, the method used by the author and his associates would still leave one sixth at the beginning, and one sixth at the end untested. And, as much as one half or more untested if the sample is taken from closer points to the center as might well happen.

Although, the tests were made on low-slump concrete suitable for paving, the large aggregate used was not the usual $1\frac{1}{2}$ in. maximum size prevailing in most of the country, but was very decidedly of the smaller dimensions, 68 per cent of which was retained on the $\frac{1}{2}$-in. and passed the $\frac{3}{4}$-in. sieve. This does not provide an ideal paving concrete in districts where large aggregate can be obtained.

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[1] Research Engineer, Ash Grove Lime and Portland Cement Co., Chanute, Kan.

[2] Chief Specifications Engineer, Parsons, Brinckerhoff, Hall and Macdonald, New York, N. Y.

[3] Consulting Engineer, Milwaukee, Wis.

I would like to see similar tests made using the Hime-Willis test to show the deviation in cement content. This test, plus sampling from the first tenth and last tenth would give more sensitive data relative to uniformity of concrete in transit mixers.

According to Fig. 9 it is to be noted that the curve indicates that for a slump of 2 in. the average discharge time is $7\frac{1}{2}$ min for 5 cu yd, so that at 10 rpm the last part of the concrete will be mixed 75 revolutions more than the first part. One would expect, especially in cool weather, a wide variation in air content, as well as in strength under these circumstances. The author's paper demonstrates the futility of adding water at the job site. This not only takes up the time of adding water, but of mixing and discharging, which can well run into a total of 20 min, and mixing at job site with low-slump concrete is one of the causes of the creation of cement balls, which are most difficult to break up with added mixing time.

MR. TIMMS.—In regard to sampling the concrete, we made a conscious effort to sample the first third of the batch soon after the start of discharge. In sampling the last third we were usually nearer the midpoint of the third in discharge than the very end. However, we had an excellent operator who warned the samplers when he could tell by the sound of the drum that the mixer was nearly discharged.

I agree with Mr. Maxon that it would have been helpful to have had the results of Hime-Willis tests to show deviations in cement content. However, when these tests were planned, the test was still in the experimental stage of development.

In reviewing the individual test loads made during the cool weather, it was noted that there was a tendency for the strength of the last third of the load to be slightly higher than the first third. However, there was no consistent relationship in the air content between the first third and the last third in spite of the longer mixing time for the last third.

# MEASURING THE RATE OF HARDENING OF CONCRETE BY BOND PULLOUT PINS*

By T. M. Kelly[1] and D. E. Bryant[1]

## Synopsis

This paper describes a method for measuring the rate of hardening of concrete. The method consists of the determination of the rate of development of bond strength. Stainless steel pins were embedded vertically in a concrete specimen immediately after the concrete was mixed. Individual pins were subsequently pulled out at increasing time intervals and the load at bond failure measured in each case. Rate of hardening curves were obtained by plotting bond strengths against time. Curves are presented which illustrate the effects of variations in concrete mixes and storage temperature upon rate of hardening.

The method for measuring the rate of hardening of concrete described in this paper, consists of determining the increase in bond strength of concrete with time. The bond strengths are determined by measuring the forces required to pull out embedded stainless steel pins at successive time intervals after contact of cement and water in the concrete mixture. The work leading to the development of the test method was initiated for the purpose of finding means for determining the setting time of concrete. It became apparent, however, as the work progressed, that in the hardening process no sharp breaking point occurs which might be interpreted as the time of set. Instead, concrete hardens at a gradually increasing rate until it reaches a definitely hard or "set" condition.

Because of the above observed relationship between time and the degree of hardening of concrete, it seemed conclusive that a test method to measure setting time is not feasible. On the other hand, it appeared that the rate of hardening of concrete under a given set of conditions can be determined, and that, perhaps, on the basis of such a determination the setting time can be arbitrarily defined. Scripture (**1**)[2] describes several of the various methods of tests for setting time which have been proposed and subjected to experimentation and discusses their limitations. He points out that the term "setting time," means something different to each individual, depending upon the specific application for which the concrete is intended. In other words, any definition of setting time must, of necessity, be arbitrary.

Whitehurst (**2**) used the soniscope to study changes occurring in the fresh concrete during its setting period by measuring the velocity of propagation of

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Vice President for Research and Senior Research Assistant, respectively, Master Builders Research Laboratories, Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1040.

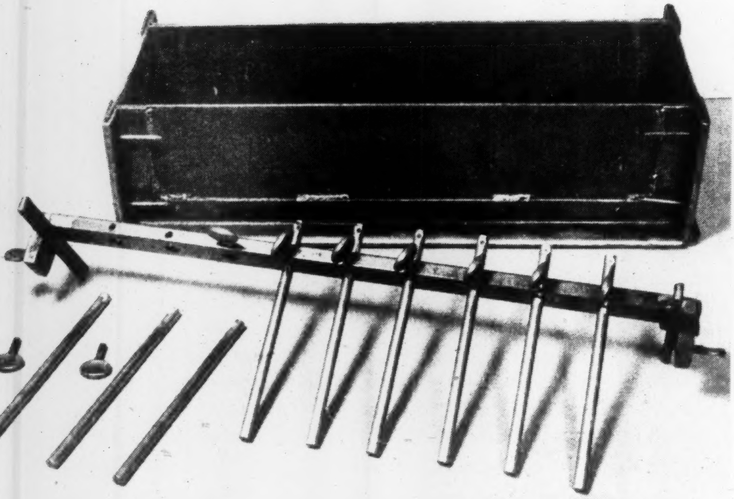

FIG. 1.—Bond Pins, Pin Holder, and Beam Mold.

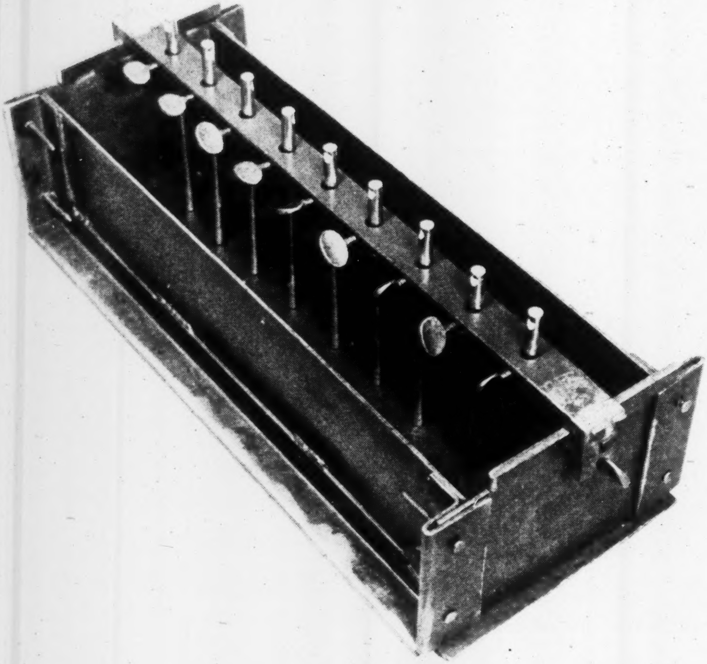

FIG. 2.—Equipment Readied for Concrete Placement.

small mechanical impulses through the concrete. He found that, during the early hours, this velocity increased at a rapid rate. After a period of time varying from $4\frac{1}{2}$ to $8\frac{1}{2}$ hr, however, the rate of increase in velocity changed suddenly, the increase continuing at a much slower pace. The point in time at which this change occurred was taken as the time of set of the concrete. Tuthill and Cordon (3) adapted the Proctor penetration apparatus to the measurement of the rate of hardening of mortar wet-screened from the concrete. They determined the relation between penetration resistance of the mortar and the vibration limit of the concrete, the vibration limit being defined as that point during hardening of concrete when it no longer can be made plastic by vibration. They stated that concrete may be considered hardened or having reached final set when the penetration resistance of the mortar, determined in accordance with their test procedure, reaches 4000 psi.

In the work reported herein there is no attempt to define or determine setting time as such. However, the method described for measuring the rate of hardening of concrete can be adapted, it is believed, to determine the time at which a given concrete under given conditions will reach a certain condition of set or hardening. Typical rate of hardening curves are presented to illustrate the general relation between time and degree of hardening of concrete. Curves are included which show the effects of temperature, air content, variations in proportioning, accelerators, and retarders on hardening rate. It is not intended that the reported effects of these variables be interpreted as applying to all concrete mixtures. It has been observed that their effects vary appreciably in magnitude, depending upon the particular combination of concreting materials used in the tests.

## TEST DETAILS AND EQUIPMENT

The component parts of the apparatus used in the rate of hardening tests are illustrated in Figs. 1, 2, and 3.

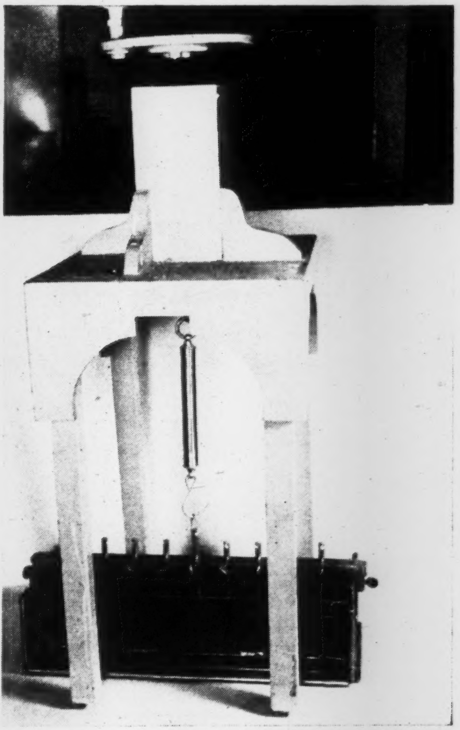

FIG. 3.—Loading Device in Position for Pull-out Test.

The general details of the method and the equipment used to obtain the test data are described below:

*Equipment:*

*Bond Pin.*—The bond pullout pins shown in Fig. 1 were cut from a $\frac{3}{8}$-in. diameter stainless steel rod. The individual pins were $7\frac{1}{2}$ in. in length. A hole was drilled perpendicular to the axis of the pin near the top, through which a ring was inserted to permit ready attachment of the loading device. The bottom ends of the pins were dressed down to remove any burrs or irregularities.

*Mold.*—The molds used in fabricating the concrete specimens for curing and storage at 50 F and 70 F were 6 in. in width, 20 in. in length, and 5 in. deep. For curing and storage at 90 F, 6-in. cube molds were used because of the limited size of high-temperature storage facilities.

*Spring Scale.*—The loads were measured by means of a Chatillon Instrument Scale, Catalog No. 0013, Fig. 3, having a capacity of 50 lb and graduated in $\frac{1}{2}$-lb increments. Its accuracy was verified by dead load tests.

*Pin Holder.*—During the fabrication of the test specimens, the pins were held in a vertical position by means of a pin holder, the lower ends of the pins being in contact with the base plate of the mold. Pins were spaced 2 in. on centers. Details of the pin holder may be seen in Figs. 1 and 2.

*Loading Device.*—Loads were applied by means of a wheel and screw device (Fig. 3) which permitted the extension of the spring scale, attached to the pin, at a steady rate of approximately 2 lb per sec.

*Preparation of Specimens:*

The pin holder was attached to the mold and the pins inserted in the holder and firmly secured in a vertical position with their lower ends resting on the base plate. Sufficient concrete was carefully distributed in the mold by means of a scoop until the mold was slightly overfull. The concrete was then compacted by means of a laboratory size, spud type, vibrator, Model 11, Serial No. 3-22655, manufactured by Viber Co. During fabrication of the test specimen, care was taken to avoid moving the pins from vertical alignment. The concrete was vibrated sufficiently to assure thorough contact with the pin surfaces, but over-

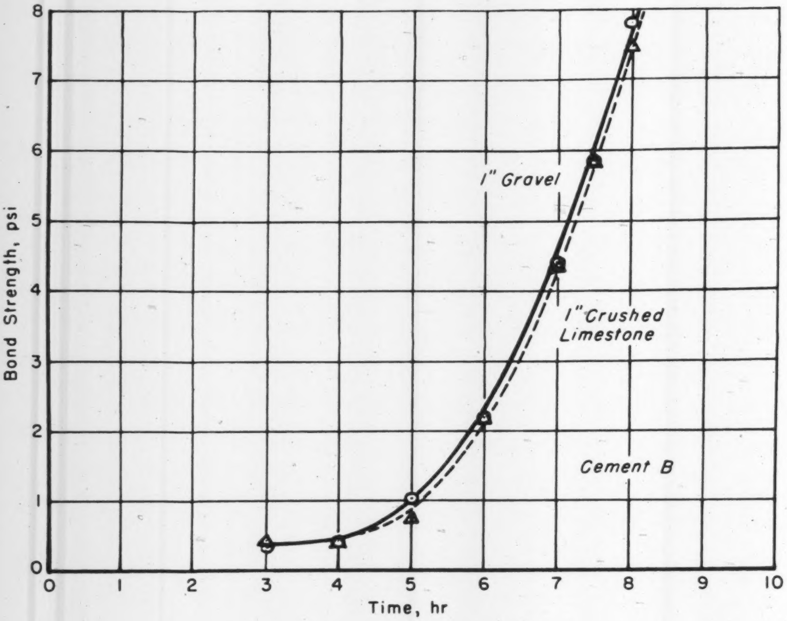

FIG. 4.—Effect of Coarse Aggregate.

vibration was avoided. Additional concrete was added as necessary so that upon completion of fabrication the mold was level full. No attempt was made to float, trowel, or strike off the surface of the specimen.

*Test Procedure:*

Just prior to pulling the first pin, the pin holder was removed, care being taken not to disturb the pins. In general, pins were pulled at approximately hourly intervals, the initial pin being pulled at an elapsed time after contact of cement and water of 3 to 4 hr. For mixes containing accelerators, the initial pin was pulled at an earlier age and additional pins at ½-hr intervals. In making a test the spring scale was attached to the top of the pin, and the load was gradually and uniformly applied keeping the line of load application coincident with the axis of the pin. The maximum load at which bond failure occurred and the time at which the load was applied, measured as elapsed time after initial contact of cement and water, were recorded.

The loads required to pull the successive pins from the concrete were converted to bond stresses by dividing the total load by the area of the embedded cylindrical surface of the pin. Rate of hardening curves were obtained by plotting these computed bond stresses as ordinates against the elapsed times as abscissae.

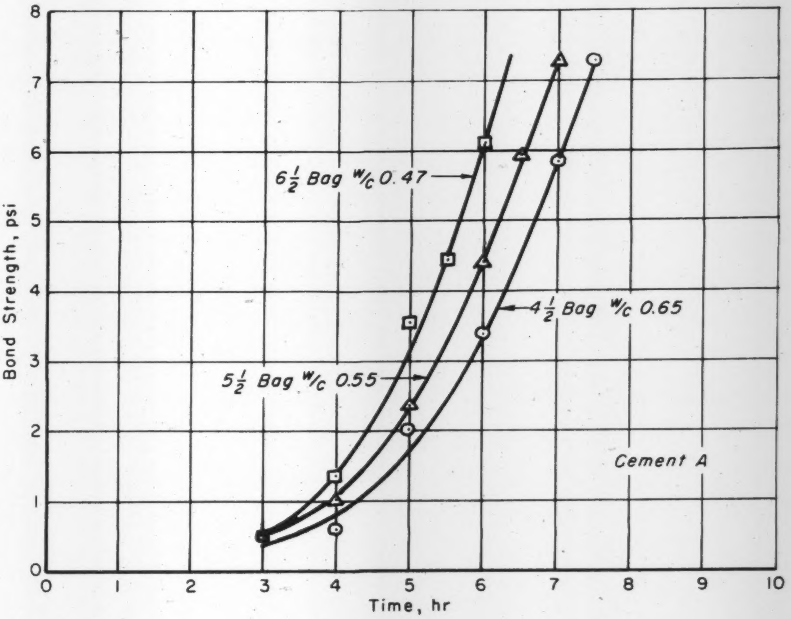

FIG. 5.—Effect of Cement Content at Constant Slump and Varying Water-Cement Ratio.

## DISCUSSION OF TEST RESULTS

The test data are shown in Figs. 4 to 13. All concrete mixes were designed in accordance with the American Concrete Inst. Standard Recommended Practice for Selecting Proportions for Concrete (ACI 613-54). Except where otherwise indicated, natural sand and 1-in. maximum size natural gravel were used. Pertinent data on the concrete mixtures are included in the following discussion:

*Effect of Coarse Aggregate:*

The two mixes shown in Fig. 4 differed only in type of coarse aggregate, with appropriate adjustment in sand-aggregate ratio. One mix contained 1-in maximum size crushed limestone and the other 1-in. maximum size gravel. The respective sand-aggregate ratios by solid volume were 0.44 and 0.41. The cement factors were constant at $5\frac{1}{2}$ sacks per cu yd. Slumps and water-cement ratios were approximately constant, being 4 in. and 5 in. and 0.56 and 0.55, respectively. Cement B, type I, was used and test specimens were maintained at 70 F. The results show that almost identical rate of hardening curves were obtained with both types of coarse aggregate.

*Effect of Cement Content at Constant Slump and Varying Water-Cement Ratio:*

The mixes shown in Fig. 5 were made with cement A, type I, and specimens were maintained at 70 F. The mixes were made to a constant slump with cement factors of 4.5, 5.5, and 6.5 sacks per cu yd, respectively. The respective water-cement ratios by weight were: 0.65, 0.55, and 0.47. The results indicate that rate of hardening increases with increasing cement content and decreasing water-cement ratio.

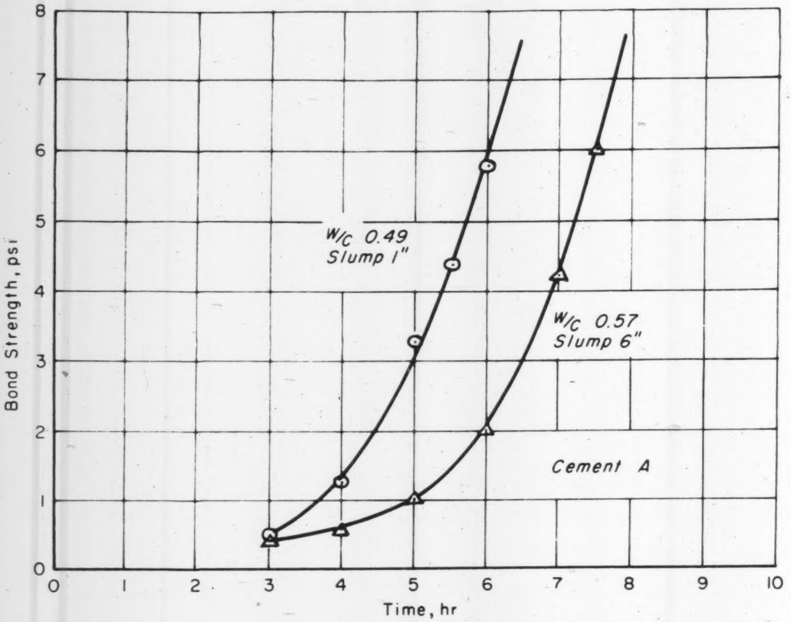

FIG. 6.—Effect of Water-Cement Ratio at Constant Cement Factor and Varying Slump.

*Effect of Water-Cement Ratio at Constant Cement Factor and Varying Slump:*

Cement A, type I, was used in the mixes shown in Fig. 6 and specimens were maintained at 70 F. The mixes were made to a constant cement factor of 5.5 sacks per cu yd with water-cement

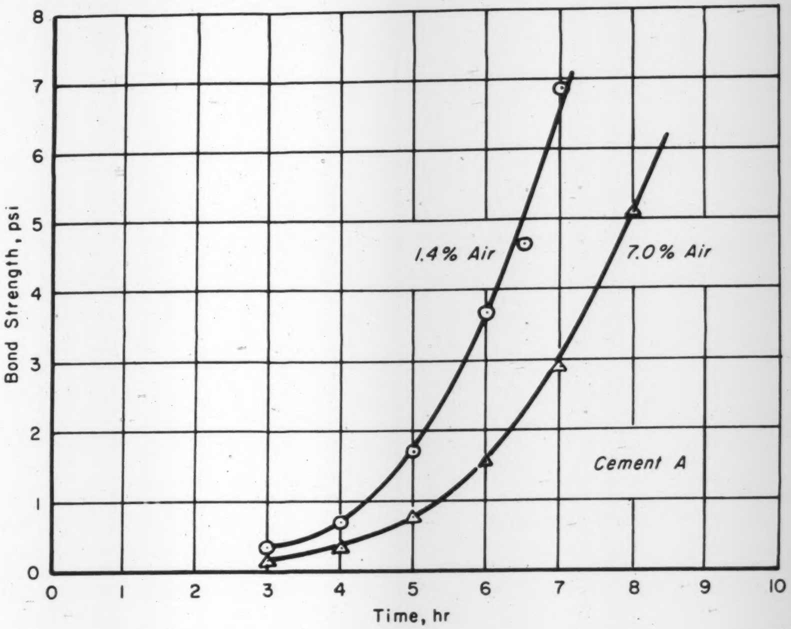

FIG. 7.—Effect of Air Content at Approximately Constant Cement Factor and Slump.

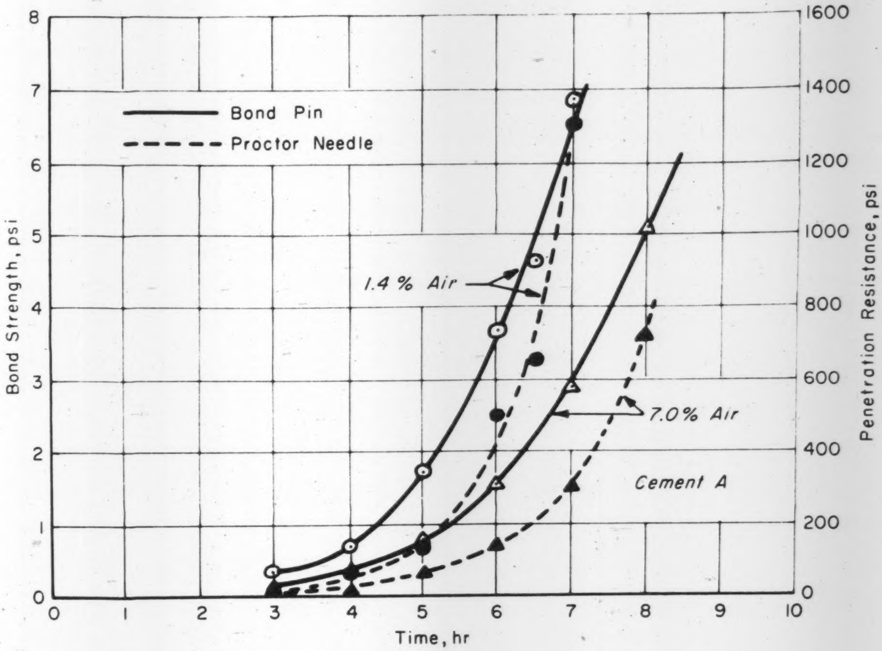

FIG. 8.—Comparison of Results of Tests by Bond Pin and Proctor Needle Methods

ratios by weight of 0.49 and 0.57, respectively. The respective slumps were 1-in. and 6-in. The data show that rate of hardening is appreciably retarded by increases in water-cement ratio and slump. The relative effects of water-cement ratio and consistency on rate of hardening may be estimated by comparing the curves in Fig. 6 with those in Fig. 5. The curves in Fig. 5 represent mixes of constant slump and varying water-cement ratio and cement factor. It appears from such a comparison that variation in consistency has approximately as great an effect on rate of hardening as variation in water-cement ratio.

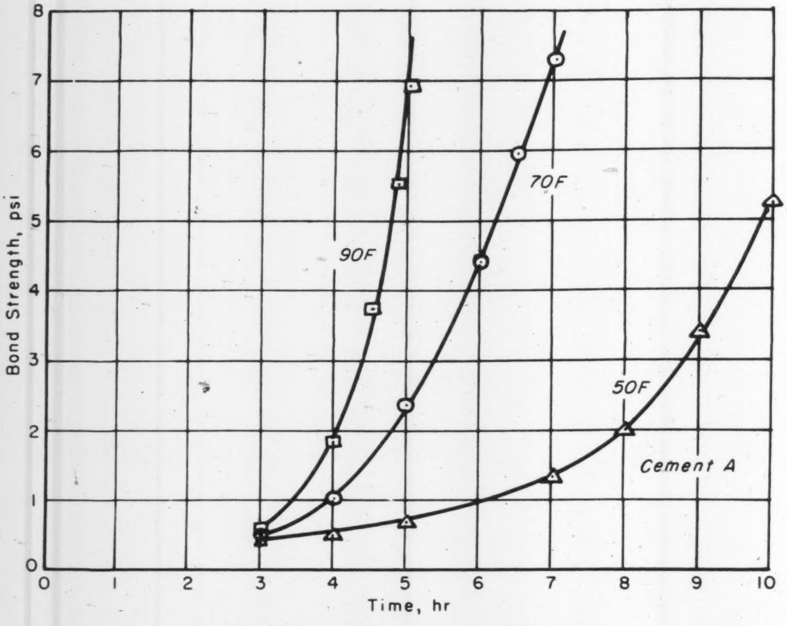

FIG. 9.—Effect of Temperature of Storage.

### *Effect of Air Content at Approximately Constant Cement Factor and Slump:*

For the mixes shown in Fig. 7, cement A, type I, was used. The specimens were maintained at 70 F. The air entrained mix had a cement factor of 5.38 sacks per cu yd; an air content of 7.0 per cent, a water-cement ratio of 0.49, and a slump of 3-in. The non-air entrained mix had a cement factor of 5.51 sacks per cu yd, an air content of 1.4 per cent, a water-cement ratio of 0.53, and a slump of 4 in. The results indicate that either the air entraining agent or the entrained air caused a retardation of the rate of hardening of the concrete.

### *Comparison of Results of Tests by Bond Pin and Proctor Needle Methods:*

Rate of hardening tests were made on mortars wet-screened from the concrete mixes discussed above in connection with Fig. 7, using the Proctor Needle penetration test method described by Tuthill and Cordon. Results obtained by both test methods are plotted in Fig. 8. It may be observed that the two test methods in this instance give similar rate of hardening curves and show approximately the same degree of retardation for the concrete in which a high percentage of air was entrained by the addition of an entraining agent. However, the two test methods differ basically in that the bond pullout test is made on the unseparated concrete mixture, while the Proctor penetration test is made on mortar wet-screened from the concrete. The fact that the bond pullout method permits determination of the rate of hardening of the actual concrete mixture, which has not been subjected to appreciable manipulation, is considered advantageous. In addition to laboratory applications, the method is adaptable to use in the field, where rate of hardening determinations could be made on the job concrete under actual conditions of placement and exposure. In limited tests to correlate vibration limit with bond strength, the vibration limit was found to occur at a point when bond strength was between 3 and 4 psi. For the particular mixes plotted in Fig. 8, the vibration limit occurred at a penetration resistance of approximately 500 psi and at a bond strength of approximately 4 psi. The vibration limit was determined on separate samples of concrete taken from the same batch used in the rate of hardening tests. These samples were placed in gallon containers and each trial for vibration limit was made on an undisturbed sample. The vibration limit was considered to be that point during the hardening of the concrete when it no longer could be made plastic by vibration.

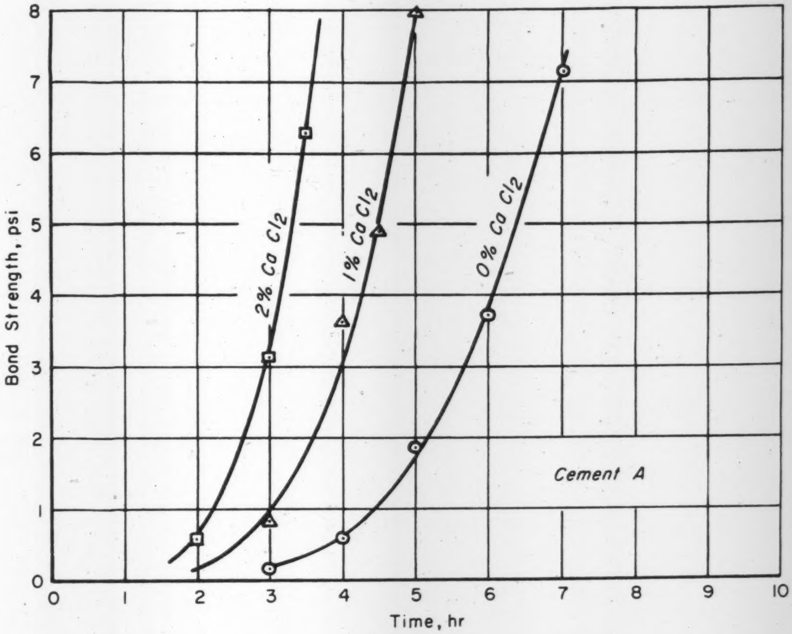

FIG. 10.—Effect of an Accelerator at 70 F.

*Effect of Temperature of Storage:*

Cement A, type I, was used in the mixes plotted in Fig. 9. Temperature of the concrete as mixed was the same for all mixes, approximately 65 F. All mixes had approximately equivalent cement factors, slumps and water-cement ratios. Immediately subsequent to specimen fabrication and for the duration of the test, the specimens were maintained at

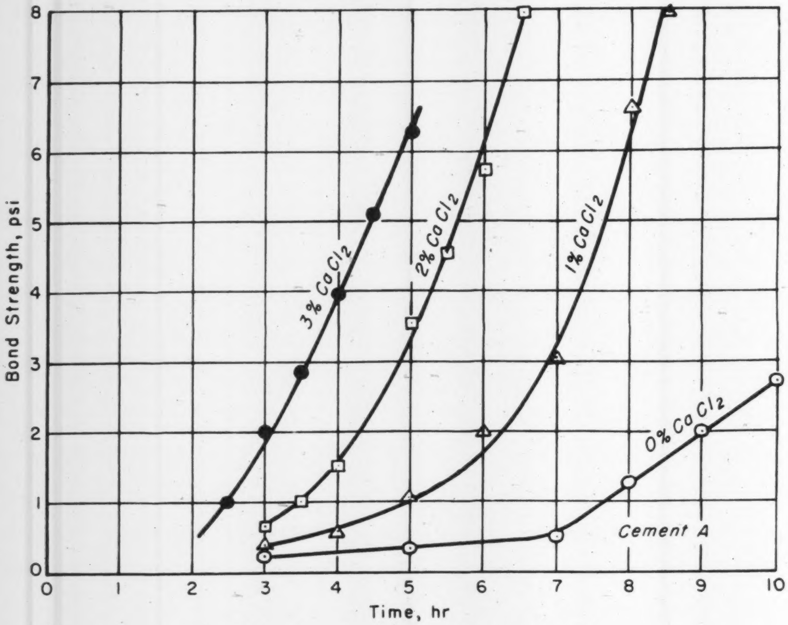

FIG. 11.—Effect of an Accelerator at 50 F.

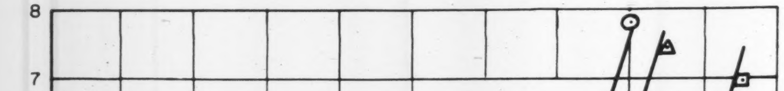

FIG. 12.—Effect of a Retarder at 70 F.

the respective temperatures of 50 F, 70 F, and 90 F. The results demonstrate the appreciable retarding and accelerating effects, respectively, of the low and high temperatures. These effects would, no doubt, have been more pronounced if the temperature of the concrete, as mixed, had been the same as the subsequent storage temperatures.

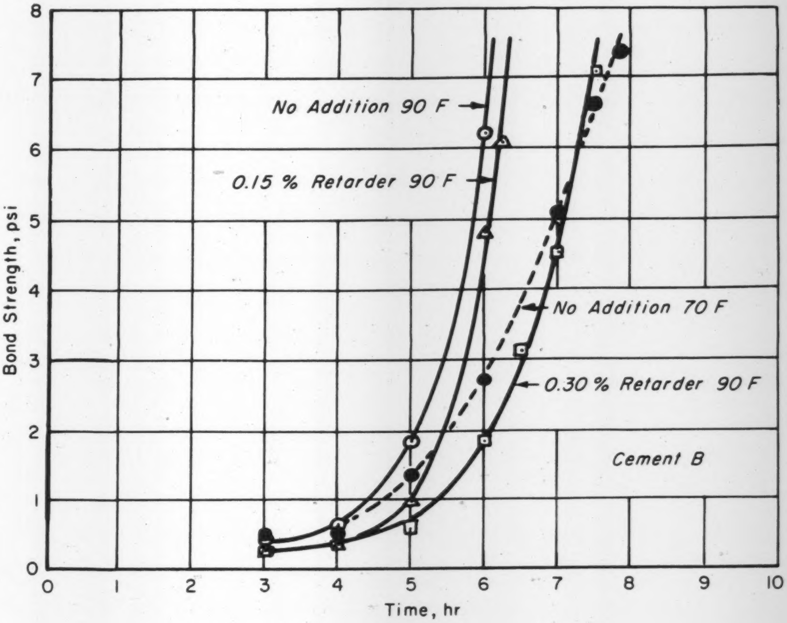

FIG. 13.—Effect of a Retarder at 90 F.

*Effect of an Accelerator at 70 F:*

Cement A, type I, was used in mixes made to constant slump and cement factor. The effects of additions of 1 per cent and 2 per cent calcium chloride by weight of cement are shown in Fig. 10. It is apparent that hardening rate is greatly accelerated by a 2 per cent calcium chloride addition at this temperature.

*Effect of an Accelerator at 50 F:*

Cement A, type I, was used in mixes made to constant slump and cement factor. Temperature of the concrete as mixed was approximately 65 F. Specimens were stored at 50 F immediately after fabrication and for the duration of the test. The effects of 1, 2, and 3 per cent calcium chloride additions by weight of cement are shown in Fig. 11. The results indicate, by comparison with Fig. 10, that a 2 per cent addition of calcium chloride gives a hardening rate at 50 F that is comparable to the hardening rate at 70 F of a mix without calcium chloride addition.

*Effect of a Retarder at 70 F:*

Cement B, type I, was used in mixes made to constant slump and cement factor. The effects of additions of a retarder at 0.15 per cent and 0.30 per cent by weight of cement are shown in Fig. 12. It is apparent that the degree of retardation varies with the quantity of retarder added.

*Effect of a Retarder at 90 F:*

Cement B, type I, was used in mixes made to constant slump and cement factor. The temperature of the concrete as mixed was approximately 65 F. Specimens were stored at 90 F immediately after fabrication and for the duration of the test. The effects of additions of a retarder at 0.15 per cent and 0.30 per cent by weight of cement are shown in Fig. 13. For purposes of comparison a mix of the same cement factor and slump, stored at 70 F, is also shown. The results indicate that the accelerating effects of high temperatures can be eliminated by the use of the retarder in the proper quantity and a hardening rate can be obtained which approximates that of a mix at 70 F containing no addition.

## SUMMARY

The rate of hardening of concrete can be measured by the bond pullout pin method of test. The degree to which rate of hardening is affected by variations in concreting materials, mix proportions admixtures, consistency, and storage temperature may also be determined. The test method has the advantage of permitting rate of hardening studies of the concrete as a whole, without removing any of its components or subjecting it to abnormal manipulation. Tests may be made with actual job-mixed concrete under conditions of placement and exposure closely approximating those prevailing on the particular job.

The data that are presented herein show that the rate of hardening of concrete is appreciably affected by changes in water-cement ratio, consistency, and storage temperature, and by the entrainment of air. It is probable that the retardation observed in the air-entrained concrete can be attributed to the chemical action of the particular organic air-entraining agent used, rather than to the presence of entrained air.

The rate of hardening curves showing the effects of variations in storage temperature, with and without the addition of an accelerator or a retarder, show that the accelerating or retarding effects of temperature upon rate of hardening may be compensated for by the use of suitable retarders or accelerators, added in the proper quantities. Such quantities, for given temperature conditions and concreting materials, can be determined by means of the test method described.

*Acknowledgment:*

The authors are grateful for the able assistance of Jack E. Moston and Otto Wm. Shimerka, who assisted in conducting the tests. In addition Mr. Moston constructed the testing apparatus.

## REFERENCES

(1) E. W. Scripture, Jr., "Setting Time," Symposium on Tests and Properties of Concrete and Concrete Aggregates, Am. Soc. Testing Mats., p. 53 (1955). (Issued as separate publication, *ASTM STP No. 169*.)

(2) E. A. Whitehurst, "Use of the Soniscope for Measuring the Setting Time of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 1166 (1951). Discussion, p. 1176.

(3) Lewis H. Tuthill and William A. Cordon, "Properties and Uses of Initially Retarded Concrete," *Journal*, Am. Concrete Inst., Nov., 1955.

# DISCUSSION

MR. ROBERT F. ADAMS[1] (*presented in written form*).—Either the bond pullout pin method described by the authors or the method utilizing the Proctor needle on mortar wet-screened from the concrete described by Tuthill and Cordon[2] provides excellent means for determining the rate of hardening of concrete. As shown by the authors of this paper and by Tuthill and Cordon, both methods which have been proposed as ASTM test methods are particularly useful in the laboratory in studying the effect of many variables such as temperature, admixtures, water-cement ratio, slump, and different cements on the rate of hardening.

Either method can also be used as a field test. Unfortunately field conditions are not subject to close control as are laboratory conditions. The engineer who uses these methods in the field for whatever purpose must be alert to any differences between the test specimens and the concrete in the structure in correlating his test results with the performance of concrete in the structure. Differences in exposure of the test specimen and concrete in the structure to sunshine or wind, differences in size and section which cause temperature differences, and varying field temperatures are some of the factors which must be recognized and skillfully controlled or interpreted to make meaningful field use of either of these methods for determining the rate of hardening of concrete.

MR. NOLAN D. MITCHELL.[3]—Have the authors tried pulling the bars from the aggregate without cement in it. They would find some factor of resistance there.

MR. T. M. KELLY (*author*).—The answer is we have not tried it, but we shall do so.

You would mean, Mr. Mitchell, to vibrate the aggregate into place?

MR. MITCHELL.—Yes.

MESSRS. T. M. KELLY AND D. E. BRYANT (*authors' closure*).—The authors are in agreement with Mr. Adams' comments regarding the interpretation of results of rate of hardening tests made under field conditions. Applicability of such test results to concrete in the structure is directly related to the degree of similarity in conditions of exposure and temperature of the concretes in the structure and specimen.

In connection with Mr. Mitchell's question regarding the resistance to pin pullout developed by a mixture containing no cement, we have obtained some data. Mr. Mitchell suggested tests with a mixture containing only the aggregates, that is, with the cement omitted. We replaced the cement component of the mixture with fly ash, however, feeling that such a mixture would more nearly approximate an actual concrete mixture and would, therefore, give a better measure of the resistance attributable to friction alone. The mixture, made with 1-in. maximum size gravel, had a slump of $4\frac{1}{2}$-in. and a fly ash content comparable to $5\frac{1}{2}$ sacks of cement per yard. Pins were pulled from the specimen at 2 hr and 4 hr after mixing. The loads required to pull the pins were essentially the same at both ages. For the eight pins pulled, the loads ranged from 0 to 1 lb. The average load was 0.4 lb which is equivalent to a bond strength of approximately 0.07 psi. These results indicate that the frictional resistance to pin movement does not significantly affect test results.

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[1] Senior Materials and Research Engineer, Department of Water Resources, Sacramento, Calif.

[2] Lewis H. Tuthill and William A. Cordon, "Properties and Uses of Initially Retarded Concrete," *Proceedings*, Am. Concrete Inst., Vol. 52, p. 273 (1956).

[3] Consultant, Arlington, Va.

# CEMENT-AGGREGATE REACTION IN CONCRETE OF A CANADIAN BRIDGE*†

### BY E. G. SWENSON[1]

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## SYNOPSIS

A study was made of a concrete bridge deck which had expanded abnormally over a period of more than 25 years. The two deck sections involved, made with different aggregates, were found to have been affected by different types of cement-aggregate reaction. One section had been seriously affected by alkali-aggregate reaction involving mainly a phyllite in the coarse aggregate, but the actual expansion and extensive deterioration of the concrete was attributed in large part to freezing and thawing under adverse moisture conditions. A feature of this case was the failure of the mortar bar test to reveal potential reactivity in the source material in spite of other evidence. Some possible explanations are considered. In the second section, abnormal expansion was probably due to the sand component which was shown to be reactive in the Conrow test, ASTM Method C 342.[2]

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An example of abnormal expansion in concrete which has occurred over a period of 25 years in a bridge deck in Eastern Canada was brought to the attention of the Division of Building Research of the National Research Council of Canada in 1953. About seven years after the deck was placed, many of the expansion joints had closed and had to be reset. Continuing "growth" of the concrete has necessitated periodic repairs, and nearly all expansion joints have now been reset at least once.

The bridge itself consists of a steel superstructure placed on masonry and concrete piers. The concrete deck, which rests on steel stringers, consists of slabs about 37 ft wide and averaging 80 ft in length. The $2\frac{1}{2}$-in. expansion joints are, for most of the length, alternated with $\frac{1}{2}$-in. "breather joints." The bridge consists of two sections, and it soon became evident that each section represented a separate case since each concrete deck had been placed by a different contractor using different sources for aggregates and cements. Records of mixes were not available.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

† This paper is a contribution from the Division of Building Research, National Research Council of Canada, and is published with the approval of the Director.

[1] National Research Council of Canada, Division of Building Research, Ottawa, Canada.

[2] Tentative Method of Test for Potential Volume Change of Cement-Aggregate Combinations (C 342 – 55 T), 1955 Book of ASTM Standards, Part 3, p. 1244.

Preliminary examination had revealed symptoms of cement-aggregate reaction in the south section, but the condition of the concrete in the north section provided no clue as to the probable cause of abnormal expansion. Since the bridge decks are exposed to extremes of weather conditions the possibility of physical as well as chemical causes had to be taken into account. As a result, it was considered necessary to carry out a more extensive series of tests than would have been necessary had there been strong evidence at the outset that one particular action was involved.

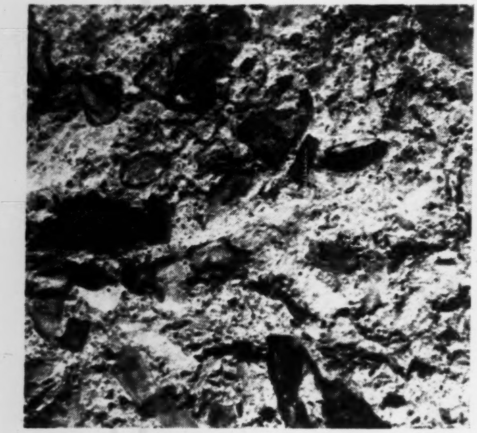

FIG. 1.—Fragment of Concrete from South Section of Bridge Deck, Showing Rims and White Deposits.

Since one possible cause was cement-aggregate reaction, cases of which have not so far been reported in Canada, it was considered imperative that expert petrographic evaluation be obtained. Fortunately, it was possible to enlist the aid of recognized authorities at the laboratories of the U. S. Bureau of Reclamation.

The present paper constitutes mainly a description of the problem and the conclusions drawn from the investigation. Considerable test data have been included, however, in the interests of those concerned with methods of test for cement-aggregate reaction. The results have certain implications that have made it advisable, for the time being, not to identify the affected structure or the sources of materials involved since these are really of local interest only.

## PRELIMINARY STUDIES

### *South Section:*

This concrete deck, 6 in. in thickness, had been placed on a floor formed with steel sheets and a paper liner which were left in place. Shortly after the concrete was placed, it was covered with a 2-in. layer of "black-top." This topping was completely replaced in 1952, reportedly as a result of the occurrence of surface depressions where the concrete had disintegrated.

Two areas were selected for coring, one considered as probably bad because of a surface depression, and the second considered to be least affected. Six corings were made, using 2- and 4-in. diameter bits. In no case was it possible to obtain intact cores, the material coming out in broken fragments which indicated an advanced state of disintegration.

Visual examination of the fragments, broken in many cases along horizontal planes, showed many aggregate particles with the broad, spongy rims usually associated with alkali-aggregate reaction. Most of the fragments were clearly fractured, and cracks and voids contained an abundance of gel-like deposits. One of the few large fragments is shown in Fig. 1; Figs. 2 and 3 show enlarged views of affected particles and gel-like deposits. That these rims were of a "pre-concrete" type was considered unlikely in view of their general appear-

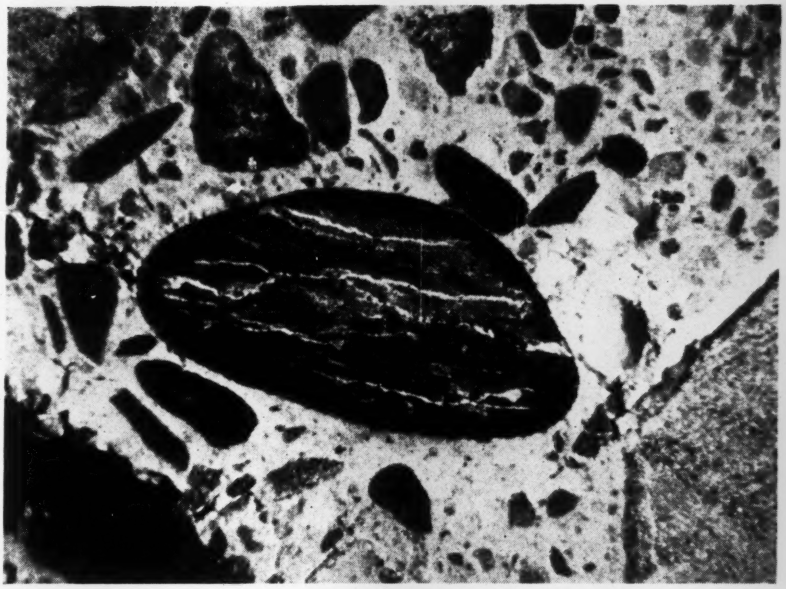

FIG. 2.—Affected Particle in Concrete from South Section, Showing Reaction Rim.

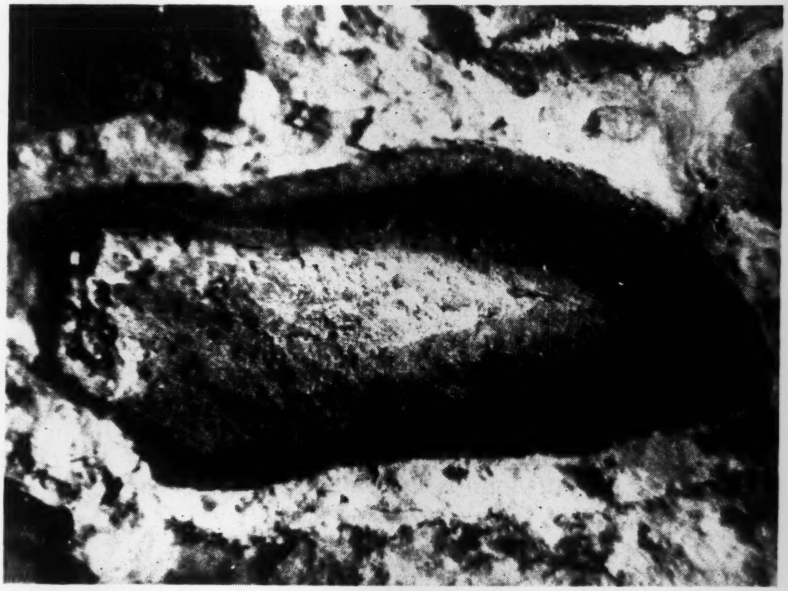

FIG. 3.—Broad Reaction Rim and White Deposits from Concrete in South Section.

ance and texture. Some of the particles were obviously of a shaley or clayey character and frequent occurrence of voids below larger particles made it probable that a fairly wet mix had been used. The "sealed" environment of the concrete in this section was such that moisture could easily be trapped. Conditions were therefore conducive not only to chemical action but also to deterioration through freezing and thawing.

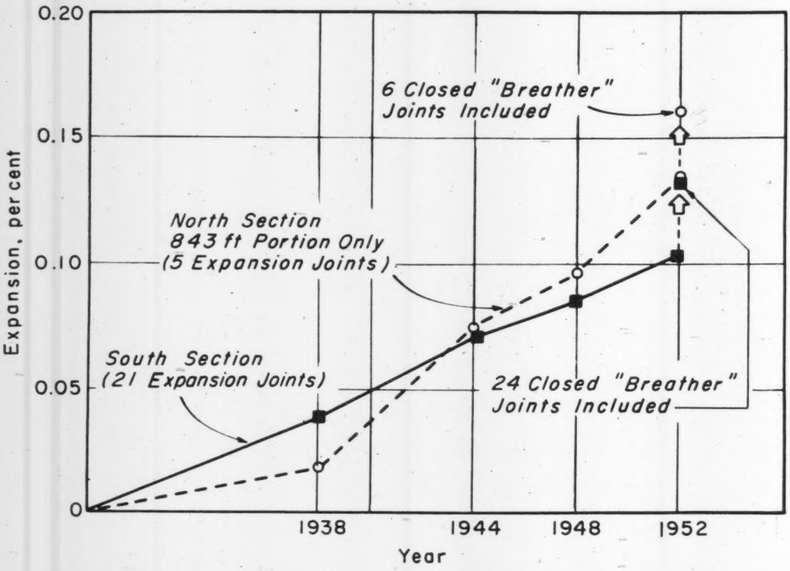

FIG. 4.—Measured Expansion of Concrete Bridge Deck-Sections.

The source of the aggregates used in this concrete was established and samples were obtained for study. The coarse aggregate had been obtained by crushing large stones and boulders from a deposit probably of fluvio-glacial origin. Much of the material was badly weathered and encrustations occurred on many rocks. A representative sample was later examined petrographically by the laboratories of the U. S. Bureau of Reclamation. Almost half of the sample was found to consist of rock types regarded as somewhat potentially reactive with high-alkali cement. These rock types included phyllites, chalcedonic sandstones, metasandstones, and somewhat chalcedonic limestones. The phyllites were soft, laminated, dark gray to black, and slightly chalcedonic. The other rock types present included sandstones, quartzites, limestones, vein quartz, crystalline limestone, schists, siltstones, claystones, and a few other miscellaneous rock types.

The sand obtained from a deposit in the same area, contained the same rock types in its coarse fractions as did the coarse aggregate. The finer fractions were composed chiefly of grains of individual minerals which were not considered deleteriously reactive with high-alkali cement. The sand as well as the stone could be regarded as physically not of first quality.

*North Section:*

The concrete deck in this section had been placed in two courses, a 6-in. layer topped with a 2-in. course, and the wooden formwork removed, leaving both upper and lower concrete surfaces exposed. Varying occurrences of random cracking were observed, the most extensive being near the joints.

Six corings were made, two from each of three widely separated areas. In all cases, the 2- and 4-in. cores were obtained intact, and the concrete appeared to be reasonably sound. The compressive strengths, taken on 4-in. cores but calculated in terms of 6 by 12-in. cylinders, averaged 5460 psi. Pulse velocities, however, averaged only 12,150 ft per sec. Attempts to detect residual expansion in some of these cores were not successful.

TABLE I.—IDENTIFICATION AND SOME PROPERTIES OF AGGREGATES USED IN TESTS.

| Aggregates | Source and Class of Material | Bulk Specific Gravity | Absorption, per cent | $MgSO_4$ Soundness, per cent loss |
|---|---|---|---|---|
| SC | Crushed boulders, from source used in south section concrete | 2.67 | 1.43 | 13.8 |
| NC | Crushed limestone, from source used in north section concrete | 2.70 | 0.42 | 8.1 |
| OC | Crushed limestone, used as unreactive reference material | 2.61 | 0.75 | 13.4 |
| SS | Natural sand, from source used in south section concrete | 2.58 | 0.64 | 14.8 |
| NS | Natural sand, from source used in north section concrete | 2.63 | 0.12 | 6.5 |
| AS | Natural sand, used as unreactive reference material | 2.47 | 0.66 | 10.0 |
| KI | Natural gravel and sand, very reactive to high-alkali cements; used as a reference material | 2.50 | 0.68 | 8.0 |

The original source of the limestone coarse aggregate used in this concrete was established, and samples were obtained for study. The quarry is located in the Palaeozoic (Lower Ordovician) formation of limestones, dolomites, and shales. A sample later examined by the laboratories of the U. S. Bureau of Reclamation was described as a somewhat carbonaceous and dolomitic limestone. Small amounts of chalcedony were detected, but this aggregate as a whole was considered not deleteriously reactive with high-alkali cement.

The sand used in the concrete on the north section may have been obtained from more than one source. At least a large portion of it was obtained from a source in the vicinity of the limestone quarry. It was a fluvial sand, the coarse fractions of which were composed predominantly of granites, gneisses, sandstones, quartzites, and other crystalline types. The finer fractions consisted chiefly of individual mineral grains. Potentially deleteriously reactive rock types, mainly phyllites, chalcedonic sandstones, and chalcedonic cherts, constituted only about 1 per cent of the sample examined. In these proportions these rock types could be disregarded. Again, both the sand and the limestone could be regarded as not of first quality.

### *Records of Expansion of the Bridge Decks:*

The actual expansion of the concrete decks had been estimated from the periodic closing and resetting of joints, and these records were made available for this investigation. These data have been plotted in Fig. 4 for the complete south section and for one part of the north section. Despite the apparent difference in the condition of the concretes in the two sections, the abnormal movement in the north section has been at least as great as that in the south section. Data for the rest of the north section were incomplete but were in good agreement with those in Fig. 4.

section, SC (stone) and SS (sand), show relatively high losses in the soundness test, as does the OC reference limestone. In addition, the absorption value for the SC crushed stone is fairly high; this is considered to have some significance in the light of later results.

The alkali contents of the three normal portland cements used in the test mortars and concretes are given in Table II. Cement M is the current counterpart of the cements MX probably used in the original concretes. The low-alkali cement E had a higher specific surface than the high-alkali M and B cements.

TABLE II.—ALKALI CONTENTS OF CEMENTS.

| Cement | Source of Data | $Na_2O$, per cent | $K_2O$, per cent | Total Alkali Calculated as $Na_2O$, per cent |
|---|---|---|---|---|
| MX.......... | Average of available data from 1926 to 1930 on cements used in affected area | 0.25 | 0.57 | 0.61 |
| M............ | From analyses of cements used in tests | 0.52 | 0.80 | 1.05 |
| B............ | | 0.56 | 0.96 | 1.19 |
| E............ | | 0.09 | 0.54 | 0.45 |

## MATERIALS USED IN COMPARATIVE STUDIES

The aggregates used for comparative tests are identified in Table I, and some pertinent properties are shown. In addition to the two sands and two coarse aggregates from the original sources used in the bridge deck concretes, several reference materials were included for control purposes. The OC reference limestone coarse aggregate is a fine-grained, somewhat fossilliferous and argillaceous limestone with a satisfactory performance record. The AS natural sand, used as a reference, also has a satisfactory field record. Two alkali-reactive gravels and sands were used as reference materials, the KI material listed in Table I and a second material designated as RR. In addition a gravel-sand material known to be reactive in the Conrow test[2] was used. This material is designated as KR.

Both the aggregates used in the south

## PHYSICAL TESTS WITH MORTAR BARS

The standard mortar bar test, ASTM Method C 227 - 52 T[3] and the Conrow test,[2] ASTM Method C 342 - 55 T were the bases for physical tests for cement-aggregate reaction with mortars. In addition, two non-standard tests on mortar bars were carried out simultaneously, primarily to determine their value in connection with the durability of aggregates. One was a wetting-drying test in which mortar bars, after moist curing for 7 days, were subjected to 18 hr of wetting at room temperature, followed by 6 hr of drying at 130 ± 3 F. At the end of 12 months, the procedure was changed to two cycles per day, 6 hr each of wetting and drying. The number of cycles obtained was approximately 120 in 6 months, 240 in 12 months, 740 in 21 months and 900 in 24 months.

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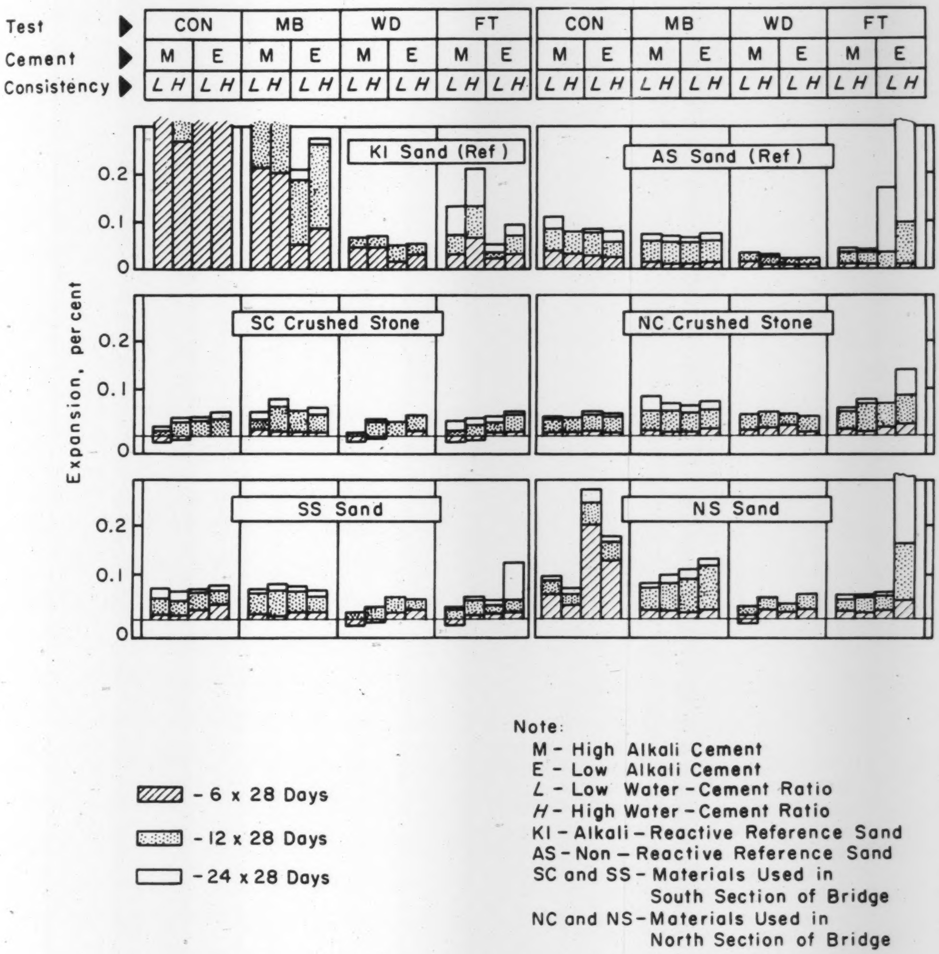
[3] Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 - 52 T), 1955 Book of ASTM Standards, Part 3, p. 47.

Duplicate specimens were used and length and weight measurements were made periodically at the end of a wetting or a thawing period. Data on the non-standard methods are included.



Note:
M - High Alkali Cement
E - Low Alkali Cement
*L* - Low Water-Cement Ratio
*H* - High Water-Cement Ratio
KI - Alkali-Reactive Reference Sand
AS - Non-Reactive Reference Sand
SC and SS - Materials Used in South Section of Bridge
NC and NS - Materials Used in North Section of Bridge

FIG. 5.—Expansion of Mortar Bars under Four Test Conditions.

The second non-standard test consisted of slow freezing in air and thawing in water. The mortar bars were moist-cured for 7 days and then air-dried in the laboratory for 21 days before being subjected to 18 hr of freezing in air to 10 F and 6 hr of thawing in water at room temperature.

For purposes of comparison, all mortar bars were made according to ASTM Method C 227 – 52 T with the following modifications: the natural sands, SS, NS and AS, were separated into six sizes (4-8, 8-16, 16-30, 30-50, 50-100, and pan) and reblended with 19 per cent of each of the first five size fractions and

5 per cent of the pan size. The stone samples, after crushing, were reblended with 20 per cent of each of the top five fractions, omitting the pan material. The latter proportions were also used for the reactive KI sand. Two mortar consistencies were used, one based on the original mortar bar test (low water-cement ratio L) and one based on the revised method (high water-cement ratio H). The water-cement ratios for the L specimens averaged 0.43 for the natural sand mortars and 0.50 for the crushed stone mortars. The corresponding values for the H specimens were 0.50 and 0.60. Mechanical mixing was employed and all bars were of nominal 5-in. length.

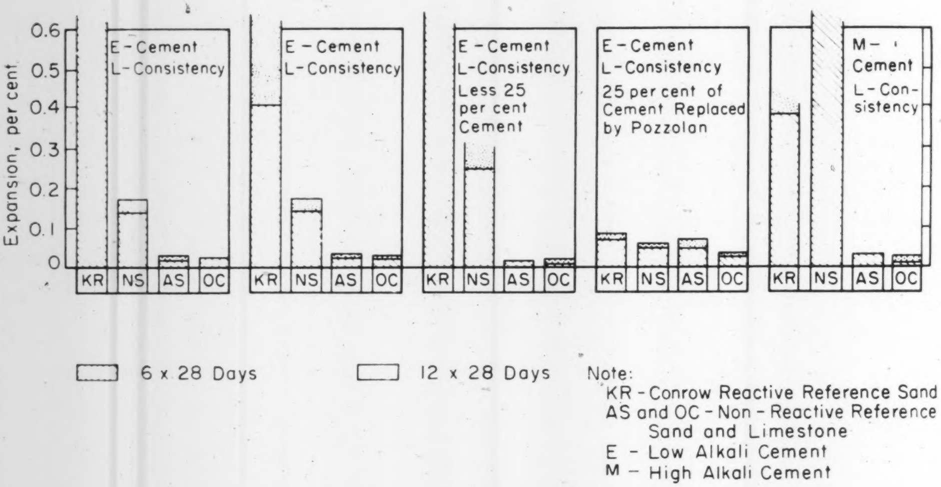


FIG. 6.—Repeat of Conrow Test on Second Sample of NS Sand.

The combined results of the mortar bar tests are shown in Fig. 5. The test conditions are designated as CON for the Conrow test, MB for the mortar bar test, WD for wetting-drying cycling, and FT for freezing-thawing cycling. The data shown correspond to periods of 6, 12, and 24 months.

Three significant conclusions may be drawn from these data: (*a*) the NS sand, which constituted at least the major portion of the sand used in the north section concrete, reacted positively to the Conrow test; (*b*) the SC stone and SS sand, which are later shown to be involved in alkali-aggregate reaction in the south section concrete, reacted negatively in the mortar bar test; and (*c*) none of the four test materials, in comparison with the reference materials, showed any unusually adverse characteristics in mortar bars subjected to wetting-drying or freezing-thawing conditions.

The Conrow test was repeated on a different sampling of NS sand. Also included was the KR reference aggregate and a pozzolan, California calcined shale. This check was considered necessary in view of uncertainty as to the nature of this type of cement-aggregate reaction and because of the serious implications concerning the current extensive use of this particular sand. Additional data are shown in Fig. 6 and generally confirm those from the original series. In Fig. 5, the higher expansion was obtained with the low-alkali cement E, but the reverse was the case with the new specimens. A replacement of 25 per cent of the cement by weight with a pozzolan was effective in controlling expansion in the mortars containing NS sand as well as those containing the KR reference sand which was known to be reactive in the Conrow test. These data may be compared with previously reported investigations into the cement-aggregate reaction for which this test was developed (**1**).[4] It is of interest to note that aggregates which react positively in the Conrow test show some reactivity in the mortar bar test.

TABLE III.—COMPOSITION AND PROPERTIES OF TEST CONCRETES.
Mix, $1:2\frac{1}{4}:3\frac{1}{2}$
Water-cement ratio, 0.60

| Composition, Cement-Stone-Sand[a] | Slump in. | Compressive Strength psi | |
|---|---|---|---|
| | | 28 days | 6 months |
| M-OC-AS........ | 3 | 4300 | 5300 |
| E-OC-AS........ | 3 | 4940 | 5100 |
| M-SC-SS........ | 4 | 3890 | 3800 |
| E-SC-SS........ | 2 | 4250 | 3970 |
| M-NC-NS........ | $6\frac{1}{2}$ | 2790 | 3400 |
| E-NC-NS........ | 5 | 3240 | 3620 |
| M-OC-KI........ | $4\frac{1}{2}$ | .... | .... |
| E-KI-AS........ | $3\frac{1}{2}$ | .... | .... |

[a] See Tables I and II.

The failure of the SC stone and SS sand to produce excessive expansion in the mortar bar test was surprising in the light of other tests which indicated definite alkali reactivity of these materials. A second series of specimens was therefore subjected to this test, in which various proportions of the SC material were combined with the unreactive OC material. This was done on the assumption, based on experimental and theoretical grounds (**2**), that only a certain range of proportions of deleterious to innocuous materials will produce excessive expansion in this test. In addition to the materials previously used, this series also included a second high-alkali cement B and a second reference sand RR, known to be alkali-reactive. In this second series the ASTM method was followed without the minor modifications previously used. After 12 months none of the specimens showed any indication of abnormal expansion except the reactive reference materials. The significance of the failure of this test is discussed later in this paper.

## PHYSICAL TESTS WITH CONCRETE BEAMS

Three non-standard tests were carried out on 3 by 4 by 16-in. concrete prisms made with materials from the sources originally used in the bridge decks. Control prisms were made with reference materials. Duplicate specimens from each batch were subjected to: (*a*) continuous conditioning at 130 ± 3 F and 100 per cent relative humidity, (*b*) wetting-drying cycling similar to the Scholer method (**3**); and (*c*) slow freezing-thawing cycling.

The composition and general properties of the test concretes are given in Table III. The sand fractions were blended in the same proportions to meet ASTM grading requirements. The coarse aggregate consisted of equal parts of each of the sizes $\frac{1}{4}-\frac{3}{8}$ in., $\frac{3}{8}-\frac{1}{2}$ in., and $\frac{1}{2}-\frac{3}{4}$ in. Aggregates were introduced in the mixes in an air-dried condition.

A number of trial mixes were made in which various proportions, consistencies, and water-cement ratios were used to obtain concretes approximating those which had been used in the bridge decks. A simplified mix was selected, using a water-cement ratio of 0.60, which was, at the time, considered a reasonable one. As a result, the differences in absorption and other properties of the aggregates produced differences in consistency and compressive strength shown in Table III. It was considered that proper allowance could be made for these differences in assessing the results of subsequent tests. Compressive strength data are based on 4 by 8-in. cylinders. The drop in strength at 6 months of the concretes made with the SC and SS aggregates (south section) was not unexpected in the light of other results with this combination of materials.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1055.

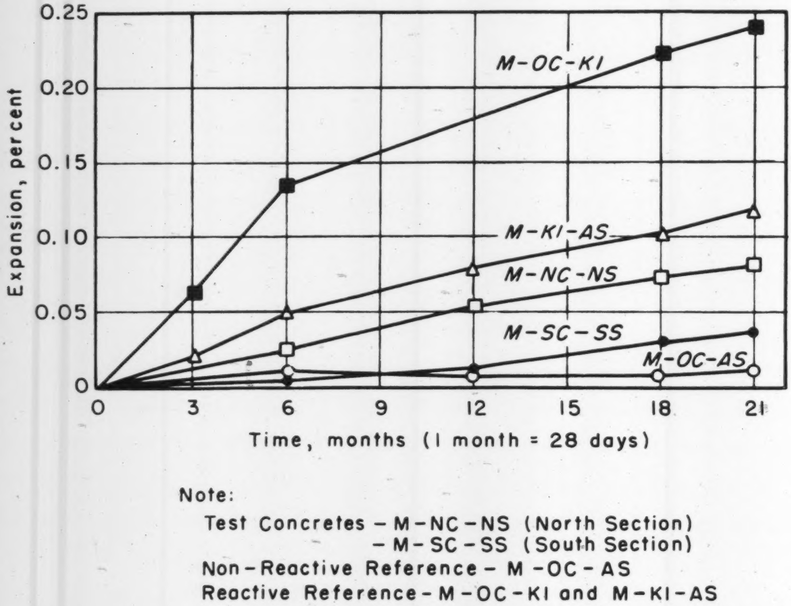

FIG. 7.—Expansion of Concrete Test Beams at 130 F and 100 per cent Relative Humidity.

The concrete beams were made with measuring studs countersunk at each end and progress in deterioration was measured by changes in length, resonant frequency modulus, and pulse velocity.

The test involving continuous conditioning at 130 ± 3 F and 100 per cent relative humidity was designed to accelerate chemical reactivity which could be measured by length change. Each beam was placed in a copper container, over but not in contact with water, then sealed and placed in an oven maintained at 130 F. Figure 7 shows the expansion of specimens over a period of 21 months. The two test specimens M-NC-NS (north section materials) and M-SC-SS (south section materials) showed more than normal expansion in comparison with the reference specimens M-OC-AS. The alkali-reactive reference specimens showed higher expansions where the reactive constituent was the sand (M-OC-KI) than in the case where the reactive component was the stone (M-KI-AS).

The wetting-drying cycling and the freezing-thawing cycling of concrete beams were carried out in the same manner as for the mortar bars. The length change data are shown in Fig. 8. They indicate that, under these conditions of cycling, there is no great difference in durability between the reference concrete and the concretes made with aggregates from the sources used in the bridge decks. The more rapid decrease in durability for all specimens under conditions of wetting and drying as compared with freezing and thawing was considered remarkable. This was not observed in the corresponding test with mortar bars, probably because the smaller specimens showed a progressive loss of water under these conditions; this was not the case with the beams. The significance of the wetting-and-drying method of rating durability of concrete, a modification of the Scholer method (3), is under continuing study in this laboratory.

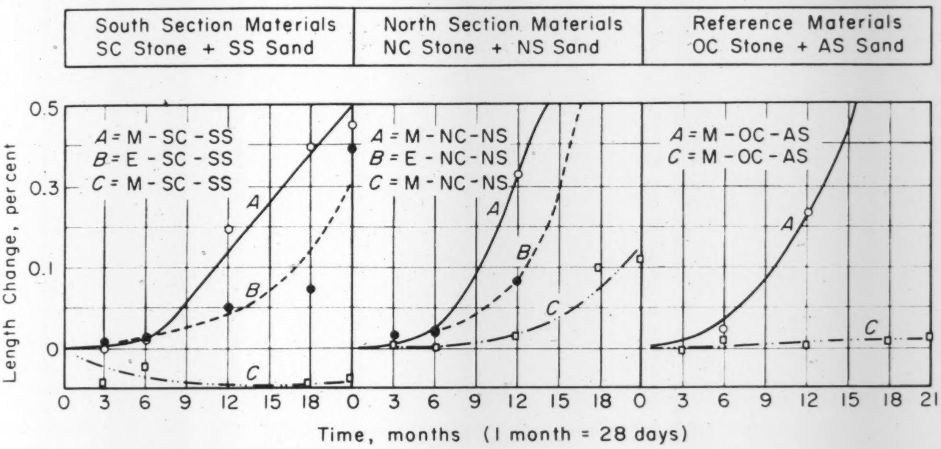

Fig. 8.—Length Change in Concrete Prisms Subjected to Wetting-Drying and Freezing-Thawing Cycling.

Curves *A* and *B* — wetting-drying
Curve *C* — freezing-thawing
Approximately 740 cycles in 21 months

The lower durability of the M-NC-NS concrete (north section) compared with the other specimens may be attributed to the lower quality as reflected by a comparison of slumps and compressive strengths (Table III). The resonant frequency moduli and pulse velocities (not shown here) were in good agreement with the length change data.

## Chemical Test for Alkali Reactivity

The quick chemical test, ASTM Method C 289 – 54 T,[5] was carried out on the four test aggregates SC, SS, NC, and NS, and on four reference materials. The data are shown in Table IV. According to the generally accepted criterion developed by Mielenz, Greene, and Benton (**4**), the SC stone was rated as deleteriously reactive to cement alkalis on the basis of this test. This stone was

TABLE IV.—DATA ON QUICK CHEMICAL TEST FOR ALKALI REACTIVITY.

| Aggregate (See Table I) | $S_c$, millimoles per liter | $R_c$, millimoles per liter | $XR_c$,[a] millimoles per liter |
|---|---|---|---|
| RR | 98.3 | 63.1 | 35.6 |
| AS | 9.7 | 70.3 | 19.9 |
| OC | 6.5 | 48.6 | 9.2 |
| SC | 86.1 | 88.9 | 42.4 |
| SS | 39.9 | 72.1 | 27.2 |
| NC | 8.0 | [b] | 17.0 |
| NS | 17.3 | 34.1 | −6.7 |
| KR | 63.2 | 34.2 | 11.8 |

[a] Modified methyl orange end point.
[b] Not done.

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[5] Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 1206.

used as coarse aggregate in the deck concrete on the south section of the bridge. None of the other test materials was shown to be reactive. The KR reference material, which was reactive in the Conrow test but not in the mortar bar test, gave a positive result in the chemical test.

## PETROGRAPHIC EVALUATION OF THE ORIGINAL CONCRETE

Detailed petrographic examination of fragments of the original concrete from the south section deck by Mr. D. Ramaley and Mr. R. C. Mielenz of the U. S. Bureau of Reclamation laboratories revealed that expansion and deterioration were caused primarily by severe alkali-aggregate reaction involving particles of phyllite, chalcedonic sandstone, and chalcedonic limestone. The opinion was expressed that other factors such as freezing and thawing may have contributed to the deterioration of the concrete. Considerable amounts of alkalic-silica gel were observed in voids and in the numerous fractures as well as quantities of calcium carbonate and calcium sulphoaluminate. Reaction rims were predominant on phyllite particles. The aggregate in the concrete samples was found to be similar lithologically to those obtained from the original sources. This case was considered to be similar to that which has occurred in the Buck hydroelectric plant (5) except that the phyllite in the SC stone was found to be less chalcedonic.

Bryant and Katharine Mather of the Corps of Engineers, U. S. Army, who later examined a sample of the affected concrete, concluded that the nature of the exposure and the poorness of quality of the concrete were primarily responsible for the deterioration, and that these factors permitted the development of an alkali-aggregate reaction which, under normal conditions, would probably be mild in degree. In their opinion chalcedony was the sole mineral reactant.

The concrete samples from the deck of the north section showed none of the usual symptoms associated with cement-aggregate reaction, and revealed no clue as to the primary cause of abnormal expansion.

## SUMMARY AND CONCLUSIONS

### *South Section:*

The deteriorated concrete in the south section of the bridge deck was seriously affected by alkali-aggregate reaction involving a phyllite present mainly in the coarse aggregate. The results of the chemical test and a petrographic evaluation of the SC stone supported the direct evidence from the petrographic examination of the concrete. This is believed to be the first case of alkali-aggregate reaction reported in Canada.

The failure of the mortar bar test to indicate potential alkali reactivity for the SC aggregate, in the face of the other evidence, suggests that the method is not entirely reliable in cases such as this, although normally this test is regarded as the most reliable of all those available. The anomaly in the present case may be due to one of several possible factors. The porosity of the aggregates, SC and SS, as reflected in part by the absorption data, may have prevented localized accumulation of gel and thereby prevented a rapid build-up of internal stresses which accompanies the formation of gel products (6). The reduction in particle size required by this test may tend to retard the reaction to a greater degree for this particular deleterious material (2). Another possibility is that the range of "pessimum" proportions is very limited for this material and is, therefore, easily missed. It would seem that the apparently anomalous behavior in this instance will not be explained until the actual constituent in the phyllite responsible for the reaction has been definitely established. In any case it was evident from the progress of expansion in the bridge deck, and also in the concrete prisms subjected to high-temperature and high-humidity conditions, that the reaction was very slow.

The evidence from petrographic examinations and laboratory tests, and from the nature of the expansion observed in this section of the deck which appeared to be increasing in rate, together with the knowledge of an inferior quality stone and a wet mix, lead to the conclusion that, under the conditions which existed, freezing and thawing were in large measure responsible for deterioration and expansion. Although an advanced case of alkali-aggregate reaction had occurred over a period of more than 25 years, abnormal expansion attributable to this phenomenon could not be reproduced in laboratory samples. It is interesting to speculate, therefore, to what extent the ultimate disintegration was due to alkali-aggregate reaction. There seems little reason to doubt that it affected indirectly, and in some degree, the observed deterioration and expansion in this section.

*North Section:*

Actual deterioration of the north section concrete was not nearly as advanced as that of the south section concrete, yet the rate and degree of expansion were at least as high. Although the wetting-drying and freezing-thawing effects in the concrete prisms indicated a low durability for this combination of materials, the slow but considerable expansion of samples under high humidity and high temperature conditions indicated that some internal reaction was the major contributing factor. In the latter case, as well as in the bridge deck concrete, expansion had apparently occurred without discernible breakdown throughout the mass of the concrete. It was concluded, therefore, that the abnormal expansion which had occurred in the north section of the concrete deck was, in all probability, attributable to the NS sand which had proved susceptible to a type of cement-aggregate reaction revealed by the Conrow test.

*Acknowledgments:*

The author is indebted to Mr. P. L. Pratley, Consulting Engineer, Montreal, for assistance in field studies and the examination of old records. Special thanks are due Mr. W. H. Price, Chief of the Engineering Laboratories, U. S. Bureau of Reclamation, for providing detailed petrographic examinations by Mr. R. C. Mielenz and Mr. D. Ramaley. Valuable advice was also received from several additional sources both in Canada and the United States.

## REFERENCES

(1) A. D. Conrow, "Studies of Abnormal Expansion of Portland Cement Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, pp. 1205–1227 (1952).

(2) T. C. Powers and H. H. Steinour, "An Interpretation of Published Researches on the Alkali-Aggregate Reaction," *Proceedings*, Am. Concrete Inst., Vol. 51, pp. 497–785 (1955).

(3) C. H. Scholer, "A Wetting and Drying Test for Predicting Cement-Aggregate Reaction," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 942 (1949).

(4) R. C. Mielenz, K. T. Greene, and E. J.

Benton, "Chemical Test for Reactivity of Aggregates with Cement Alkalis; Chemical Processes in Cement-Aggregate Reaction," *Proceedings*, Am. Concrete Inst., Vol. 44, pp. 193–221 (1948).

(**5**) H. A. Kammer and R. W. Carlson, "Investigation of Causes of Delayed Expansion of Concrete in Buck Hydro-electric Plant," *Proceedings*, Am. Concrete Inst., Vol. 37, pp. 665–671 (1941).

(**6**) D. McConnell, R. C. Mielenz, W. Y. Holland, and K. T. Greene, "Cement-Aggregate Reaction in Concrete," *Proceedings*, Am. Concrete Inst., Vol. 44, pp. 93–128 (1948).

# FALLACIES IN THE CURRENT PER CENT OF TOTAL ABSORPTION METHOD FOR DETERMINING AND LIMITING THE MOISTURE CONTENT OF CONCRETE BLOCK*

By Carl A. Menzel[1]

## Synopsis

Information from various sources shows that cracking in concrete masonry walls can be reduced by minimizing moisture shrinkage in the wall. To do this it is necessary to preshrink the units by adequate drying before they are placed in the wall. The degree of dryness needed will vary, but for any given locality the concrete should be in an air-dry condition corresponding to the average annual relative humidity of the outside air for that locality. This principle applies to all types of concrete masonry units regardless of type of aggregate or method of curing used.

The long-standing test for moisture content described in ASTM Method C 140 – 56[2] was intended to limit the permissible moisture content of concrete block. However, it is inadequate since it fails to give a significant and realistic indication of the moisture condition of the concrete such as is needed to show whether or not block are at the desired degree of dryness to reduce drying shrinkage and cracking in the wall.

This paper presents data showing that the moisture held by air-dry concrete at equilibrium with a given relative humidity is influenced by many factors and is not significantly related to the total absorbed water in a saturated block. Hence, the current Per Cent of Total Absorption Method should be replaced as soon as possible by the Relative Humidity Method which provides a reliable indication in about 20 to 60 min of the state of dryness and potentiality for drying shrinkage of concrete block in subsequent exposure to atmospheric conditions. This is less than 2 per cent of the 48 to 72 hr needed to saturate and then oven-dry block to constant weight as required by the current ASTM method.

For about 25 years the ASTM and Federal Specifications for Concrete Block have had provisions which limit the permissible moisture content of the concrete at the time of delivery to the job site. The primary purpose of this moisture content limitation is to reduce the shrinkage which occurs in individual units upon drying to an air-dry condition. This drying shrinkage tends to produce cracks in concrete masonry walls which should be minimized as far as practical.

A discussion of general considerations of the cracking problem in concrete masonry walls is given in Appendix I.

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[2] Methods of Sampling and Testing Concrete Masonry Units (C 140 – 56), 1956 Supplement to Book of ASTM Standards, Part 3, p. 511.

This indicates why it is necessary to minimize moisture shrinkage in the wall by preshrinking the masonry units by adequate drying *before* they are placed in the wall. The degree of dryness needed will vary, but for any given locality the concrete should be in an air-dry condition corresponding to the average annual relative humidity of the outside air for that locality. This principle applies to all types of concrete masonry units regardless of type of aggregate or method of curing used.

At this time there are two methods available for determining and expressing the moisture condition of concrete block:

1. The long-standing ASTM method[2] in which moisture content is expressed in per cent of total absorption by the ratio:

$$\frac{\text{weight of moisture in block as sampled} \times 100}{\text{weight of moisture in block when saturated}}$$

$$= \frac{\text{weight as sampled—oven-dry weight} \times 100}{\text{saturated weight—oven-dry weight}}$$

2. The Relative Humidity Method in which the moisture condition of the hardened concrete is expressed directly in per cent of relative humidity. The development of this method began in November 1947 and is described in a paper by Menzel (**1**).[3]

The relative humidity method is on a sound scientific basis and provides a reliable indication in about 20 to 60 min of the degree of dryness and potentiality for drying shrinkage of concrete block in subsequent exposure to atmospheric conditions. This is less than 2 per cent of the 48 to 72 hr needed to saturate and then oven-dry block to constant weight as required by the ASTM method C 140[2] for moisture content and absorption.

Moreover, the per cent of total absorption method, based on weight loss during oven drying, and expressed in per cent of total absorption, is not on a sound scientific or realistic basis for minimizing drying shrinkage. The outstanding fallacy implicit in this method is that a given state of dryness can be expressed as a fixed percentage of the total amount of water that the material is able to hold when saturated. The truth is that the state of dryness is manifested by the relative humidity found within the pores of the concrete. The principal purpose of this paper is to present data demonstrating that truth and why the current ASTM method should be replaced by the relative humidity method. It is assumed that the reader is familiar with the theory, development and application of the relative humidity method as described in reference (**1**).

## FACTORS INFLUENCING AMOUNT OF WATER HELD BY HARDENED CEMENT PASTE AT VARIOUS RELATIVE HUMIDITIES

In common with many materials, hardened concrete has a natural tendency to lose or absorb moisture with changes in the moisture content or relative humidity of the air. Concrete that is obviously wet or damp loses a considerable portion of its moisture before it approaches an air-dry condition—a state of approximate balance or equilibrium with the average relative humidity of the surrounding air.

A piece of concrete loses or gains water and shrinks and swells with changes in relative humidity largely because the hardened cement paste which binds the aggregate particles together responds to humidity changes. The aggregate in the concrete is relatively inert.

It may be pointed out that, although the aggregate is relatively inert and the paste is the primary shrinking component of the concrete, the aggregate offers a considerable degree of restraint to the shrinkage of the paste. Hence, other things being equal, a concrete made with a strong relatively incompressible aggregate will shrink appreciably less than a concrete made with a weak, more compressible aggregate.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1070.

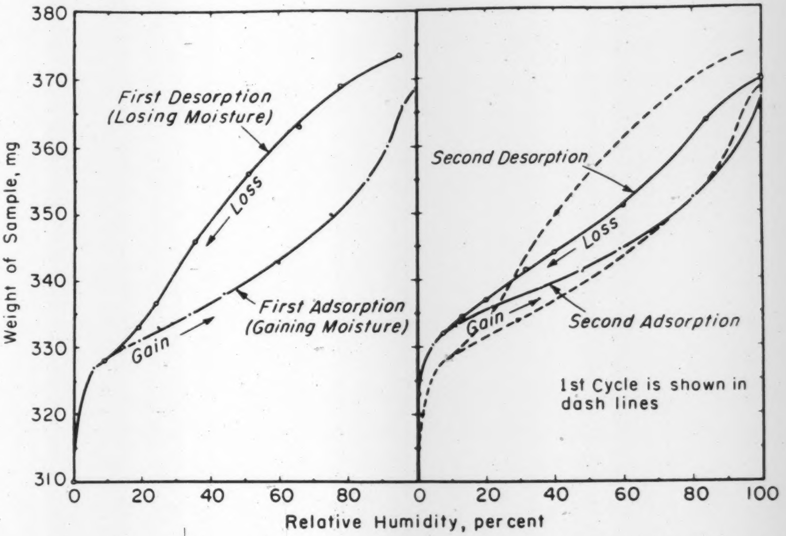

FIG. 1.—Effect of Desorption and Adsorption Cycle on Amount of Water Held by Paste at any Given Relative Humidity.

Data from Figs. 2 to 6A and 2 to 6B of reference (3). Water-cement ratio of paste = 0.50.

Excellent and reliable data on the physical properties of hardened portland cement paste have been reported by T. C. Powers and T. L. Brownyard (2) and were used in the discussion which follows on the effect of various factors which influence the amount of water held by the cement paste at various relative humidities. More recently, related data on this subject have been reported by George Verbeck (3), N. C. Ludwig and S. A. Pence (4), and Joseph J. Shideler (5) and were also used in the discussion.

*Effect of Desorption and Adsorption:*

The curves of Fig. 1 are based on tests in which a hardened cement paste sample was exposed to several cycles of decreasing and increasing relative humidities. They show that the amount of water held at equilibrium at any given relative humidity may vary substantially depending on whether the paste has been losing moisture to the atmosphere (a process called desorption) or has been taking up moisture from the atmosphere (a process called adsorption). The curves show that more moisture is held at a given relative humidity during a desorption cycle than the next adsorption cycle. There is also a difference between the water held during the first and second desorption or adsorption cycles. Corresponding influences of desorption and adsorption are shown in Fig. 5 based on tests with concrete block and will be discussed later.

*Effect of Length of Curing Period:*

The curves in the left diagram of Fig. 2 show that the amount of adsorbed water held by the paste at a given rela-

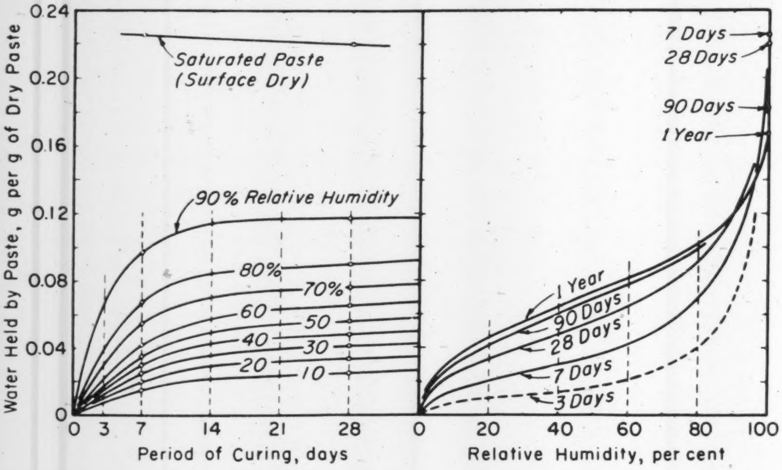

FIG. 2.—Effect of Length of Curing Period on Amount of Adsorbed Water Held by Paste at any Given Relative Humidity.

Data from Figs. 2 to 11 of reference (**2**). Water-cement ratio of paste = 0.31.

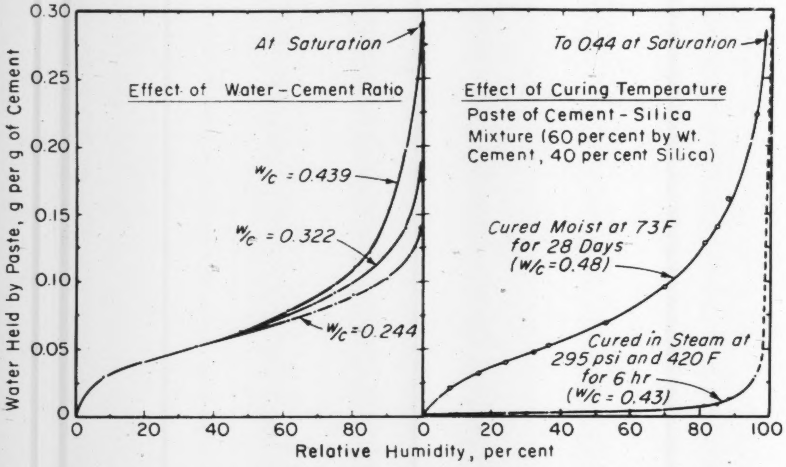

FIG. 3.—Effect of Water-Cement Ratio and Curing at High Temperature on Amount of Adsorbed Water Held by Paste at any Given Relative Humidity.

Data from Figs. 2 to 14 and 2 to 24 of reference (**2**).

tive humidity increases with the length of the curing period. The right diagram shows that the amount of adsorbed water held at 40, 50, 60, or 70 per cent relative humidity is doubled when the curing period is increased from 3 days to 7 days, tripled when it is increased to 28 days and nearly quadrupled when moist curing is extended to 1 yr. Note also that the water held by the saturated paste at 100 per cent relative humidity, decreases with duration of the moist curing period. These data show that if two similar blocks are cured differently, they will hold different amounts of water when they are at an equal state of dryness. The per cent of total absorption method would indicate, falsely, the poorer block to be drier than the better one.

*Effect of Water-Cement Ratio of Paste:*

The curves in the left diagram of Fig. 3 are based on tests with pastes ranging in water-cement ratio from 0.244 to 0.439 cured to equal states of cement-hydration. They show that the amount of adsorbed water held by the paste at a given relative humidity was about the same below 50 per cent relative humidity, but at relative humidities from 50 to 100 per cent increased gradually with the water-cement ratio of the paste. Note that the water held at saturation at 100 per cent relative humidity was about twice as much for the paste with a water-cement ratio of 0.439 as for the paste with water-cement ratio of 0.244. These data indicate that with pastes of appreciably higher water-cement ratios as found in the lean concrete mixes normally used in concrete block the effect of variations in water-cement ratio can be expected to be substantial and particularly at high relative humidities and saturation. This means that block made with pastes having different water-cement ratios will contain different amounts of water at equal states of dryness, yet the per cent of total absorption method would indicate them to have different states of dryness.

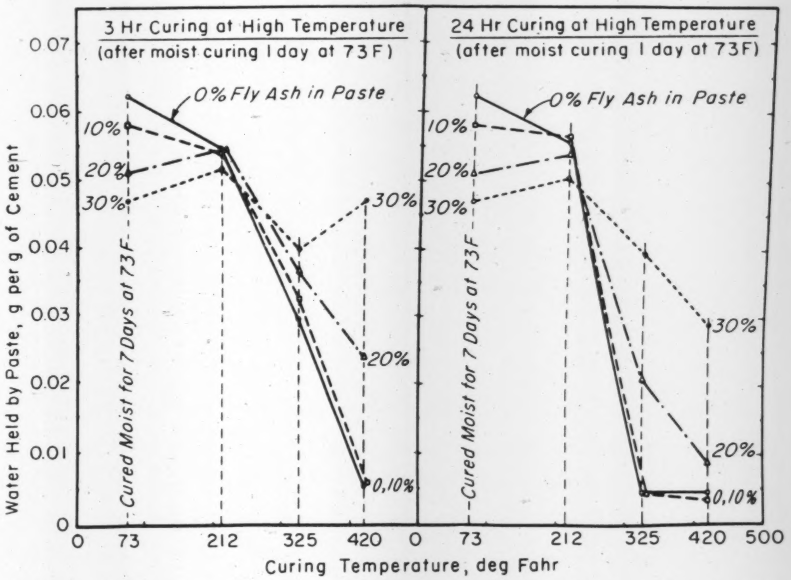

FIG. 4.—Effect of Temperature and Duration of Curing and of Fly-Ash on Adsorbed Water Held by Pastes at 36 per Cent Relative Humidity.

Data from Table VI of reference (**3**). Water-cement ratio (by weight) = 0.40, 0.44, 0.50 and 0.57, respectively, for 0, 10, 20, and 30 per cent fly-ash in paste.

*Effect of Curing at High Temperature:*

The curves in the right diagram of Fig. 3 show that the amount of adsorbed water held at a given relative humidity is markedly less (only 5 to 13 per cent as much) when the paste (cement-silica mixtures) is cured in high-pressure saturated steam at 295 psi and 420 F for 6 hr than when it is cured moist for 28 days at 73 F. Further data on the effect of curing temperature will be discussed in connection with Fig. 4.

### *Effect of Temperature and Duration of Curing, and of Pozzolanic Material:*

Powers and Brownyard (2) have shown that the adsorbed water held at 36 per cent relative humidity is directly proportional to the extent of hydration and the internal surface area available to water vapor in the hardened paste. Moreover, this internal surface area is an indicator of the relative amount of adsorbed water that would be held by a hardened paste at various relative humidity levels above 36 per cent and is related to drying shrinkage. More recently, Verbeck (3) has not only obtained additional data indicating that the amount and surface area of the hydrated cement gel is closely related to drying shrinkage but also that it is closely related to carbonation shrinkage, degree of carbonation, modulus of elasticity, and the water permeability of the paste.

In Fig. 4 the data from Verbeck's tests show the substantial effect of temperature, duration of curing and of fly-ash (a pozzolanic material) on the adsorbed water held by pastes at 36 per cent relative humidity instead of a wide range of humidities shown in previous figures. It should be noted that at 73 F all specimens were cured moist for 7 days but that at 212, 325, and 420 F specimens were cured in saturated steam for 3 hr or 24 hr after moist curing 1 day at 73 F.

Since the graphs show that the amount of adsorbed water held at 36 per cent

TABLE I.—EFFECT OF VARIATIONS IN GRADING OF AGGREGATE ON TOTAL ABSORBED WATER AT SATURATION AND ON THE INDICATED MOISTURE CONTENT OF AIR-DRY BLOCK WHEN EXPRESSED IN TERMS OF TOTAL ABSORPTION.

Values in columns 1 to 5 were taken from Tables I and XXVII in a Portland Cement Assn. report by Menzel on Tests of the Fire Resistance and Strength of Walls of Concrete Masonry Units. Jan., 1934. Values in column 6 represent approximate weight of absorbed water based on values in columns 4 and 5. Values in column 7 represent approximate moisture contents, in per cent of total absorption, if the moisture in the cement paste of each block was assumed to be constant at 1.0 lb per block. This assumption is warranted, for the sake of illustration, when all block in a given series are at the same air-dry condition at equilibrium with the same relative humidity.

| Test Number | Fineness Modulus | Cement per Block, lb | Weight of Air-Dry Block, lb | Total Absorbed Moisture | | Moisture Content, per cent of Total Absorption |
|---|---|---|---|---|---|---|
| | | | | Per cent by weight | lb per Block | |
| *1* | *2* | *3* | *4* | *5* | *6* | *7* |
| | | | SAND AND GRAVEL | | | |
| 6 | 4.50 | 4.43 | 49.9 | 5.0 | 2.50 | 40 |
| 7 | 4.00 | 4.39 | 48.5 | 6.1 | 2.96 | 34 |
| 8 | 3.50 | 4.20 | 46.6 | 7.3 | 3.40 | 29 |
| 9 | 3.00 | 4.01 | 44.3 | 7.9 | 3.50 | 29 |
| 64 | 2.00 | 4.39 | 41.2 | 10.3 | 4.25 | 24 |
| | | | HAYDITE | | | |
| 59 | 4.75 | 4.40 | 23.8 | 12.0 | 2.85 | 35 |
| 60 | 4.25 | 4.45 | 25.6 | 14.1 | 3.61 | 28 |
| 61 | 3.75 | 4.41 | 27.2 | 17.2 | 4.69 | 21 |
| 62 | 3.00 | 4.43 | 28.2 | 20.3 | 5.72 | 18 |
| 63 | 2.07 | 4.47 | 28.5 | 22.3 | 6.36 | 16 |

relative humidity was greatly influenced by the temperature and duration of curing and the use of 10, 20, and 30 per cent of fly-ash in the paste, these factors can be expected to have a corresponding influence on the amount of adsorbed water held at higher levels of relative humidity, not only in the cement paste but in corresponding concrete in block. Similar results regarding the influence of curing temperature on surface area are reported in Fig. 6 of the paper by Ludwig and Pence (4) in which the curing period was for 7 days at all temperatures.

The data in Fig. 4 for the temperature range 73 to 212 F cover moist curing in steam at atmosphere pressure, and the data for the temperature range of 212 to 420 F cover curing in saturated steam at a gage pressure from 0 to 295 psi. In the usual cycle of autoclave curing at a block plant, the block are exposed to saturated steam at a temperature of 350 to 365 F and at a gage pressure of 120 to 150 psi for 7 or 8 hr. Hence, the data in Fig. 4 embrace variations in curing conditions encountered in the production of concrete block. These variations can be expected to have similar effects on the amount of water held by the cement paste in concrete block.

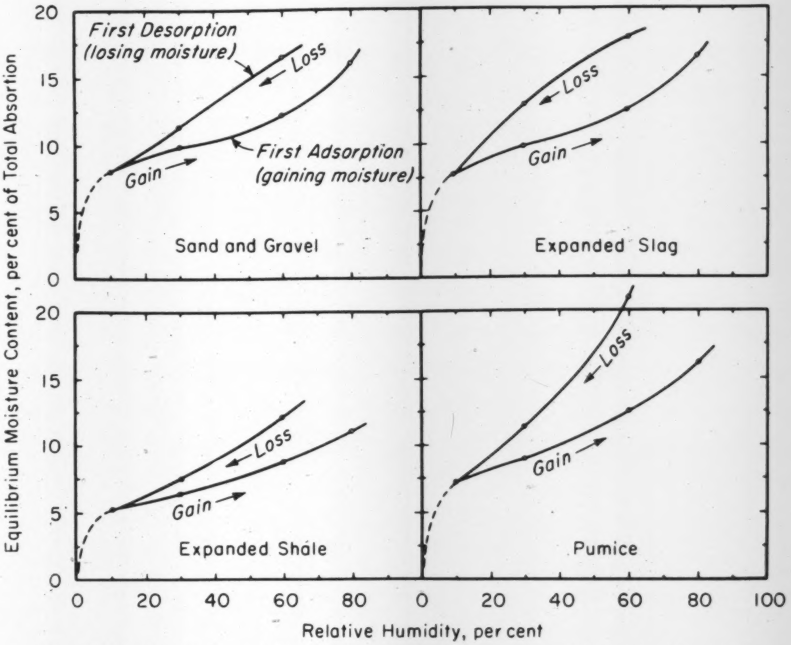

FIG. 5.—Effect of Desorption and Adsorption Cycle on Amount of Water Held by Different Block Concretes at Various Relative Humidities.

Data from Table VII of reference (5).

*Effect of Carbonation:*

Tests reported by Verbeck (3) indicate that the adsorbed water held by cement paste at 36 per cent relative humidity is increased by carbonation. This means that carbonation increases the internal surface area of the paste and that carbonation can be expected to increase the amount of adsorbed water held by the paste at higher levels of relative humidity.

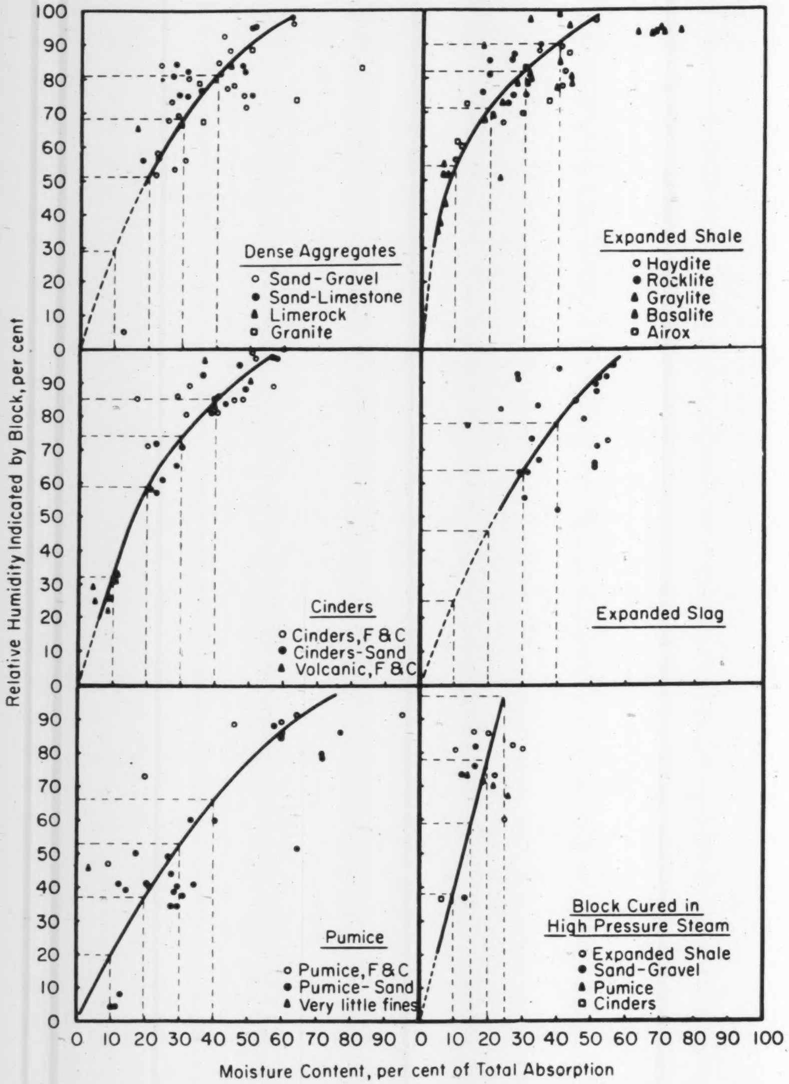

FIG. 6.—Relative Humidity *versus* Moisture Content, in per Cent of Total Absorption, Indicated by Block made with Various Aggregates.

Data are from Table III and Fig. 7 of reference (**6**) and are based on tests of 200 block at 16 plants at various locations in the United States. Block were taken from stock piles at each plant.

## FACTORS INFLUENCING WATER HELD IN CONCRETE AT VARIOUS RELATIVE HUMIDITIES AND AT SATURATION

It will be apparent that water held at various relative humidities by the concrete will not only be influenced by the adsorption properties of the paste but by the cement content or amount of paste in the concrete. It may also be influenced to some degree by the adsorption properties of the aggregate.

Moreover, the amount of water in

saturated concrete will depend on the porosity and amount of paste, the porosity of the aggregate, and the volume of voids in the lean concrete as affected by gradation of aggregate.

The values in column 6 of Table I show the effect of variations in grading of aggregate on total absorbed water at saturation. These data, like those already cited, show how units that are actually equally dry appear to be at different states of dryness when tested by the per cent of total absorption method. The values in column 7 show that the indicated moisture content, in per cent of total absorption, varies from 24 to 40 per cent for sand and gravel block and from 16 to 35 per cent for Haydite block. Note that block with the highest absorption, and hence less desirable than block with lower absorption, appear to be drier and less likely to shrink because their indicated moisture content is lower, but this is not true.

## RESULTS OF TESTS ON CONCRETE BLOCK

### *Factors which Influence Moisture Held by Paste Appear to Influence Moisture Held by Concrete:*

The desorption and adsorption curves of Fig. 5 are based on tests with concrete block by Shideler (5). They show that the effect of desorption and adsorption cycles on the amount of water held by different block concretes at various relative humidities is very similar to the effect shown by the curves in Fig. 1 for cement paste alone. This similarity between the effects of adsorption and desorption of the paste and of concrete is a good indication of the effect of the paste on the water held by the concrete at various relative humidities. It affords a basis for expecting that the other factors such as the length of curing period, water-cement ratio, curing temperature, etc., which influence the water held by the paste alone influence the amount of water held by concrete block in a similar manner when the cement paste in them is exposed to these factors.

In Fig. 5 the moisture content of the block is plotted in per cent of total absorption and for a given block represents the relative weight of moisture in the block since the water at saturation is constant. Note that at 60 per cent relative humidity the moisture content at desorption is substantially higher than at adsorption by about the following percentages: 36 for sand and gravel; 37 for shale; 42 for slag; and 68 for pumice. Also that at 60 per cent relative humidity the block made with different aggregates had moisture contents between 12 and 21 per cent (of absorption) during desorption and between about 9 and 12.5 per cent during adsorption, whereas the curves of Fig. 6 indicate a range of 12 to 34 per cent for the same four types of aggregate. It will be evident from these tests alone that there is no significant relation between the moisture content of concrete block in per cent of absorption and relative humidity, even for a specific block, because of differences in actual moisture content between desorption and adsorption.

### *Relative Humidity versus Moisture Content in Per cent of Total Absorption:*

Figure 6 shows the results of extensive field tests (6) on the moisture content of 200 concrete block as found at 16 plants at various locations in the United States. In these tests whole 8 by 8 by 16-in. block samples were divided into two approximately equal portions so that the moisture condition of one portion by the relative humidity method could be compared with the moisture condition of the other portion by the per cent of total absorption method.

The results plotted in each diagram are characterized by a wide scatter of points and the curve drawn through them is one of several which may be drawn in an attempt to indicate a relationship between the two values of moisture condition. However, no close relationship is indicated in any diagram for a given aggregate. The curves differ substantially for the different aggregates and indicate that for a given relative humidity the moisture content in per cent of absorption is lowest for expanded shale and increases progressively with cinders, dense aggregates, expanded slag and is highest with pumice-sand. It appears possible that these differences may be influenced by differences in the adsorption properties of the aggregate, but this remains to be established by more direct methods.

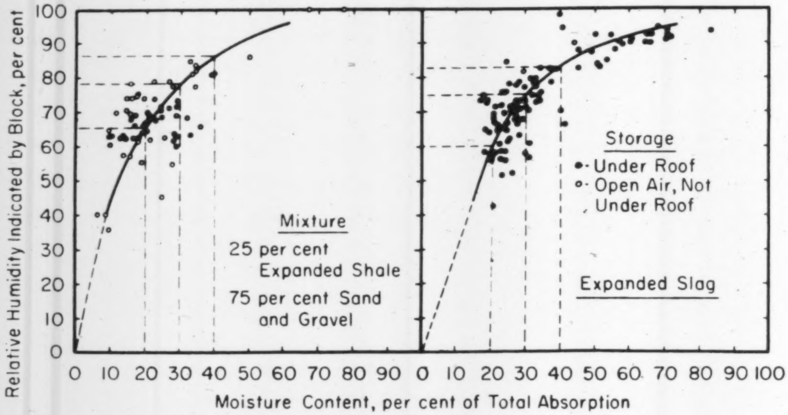

FIG. 7.—Relative Humidity *versus* Moisture Content, in per Cent of Total Absorption, Indicated by Block Tested at two Locations.

Left diagram is same as Fig. 3 of reference (**6**) and based on tests with relative humidity apparatus No. 8 at Fort Lewis Materials Laboratory, Corps of Engineers, of 69 block stored under cover at jobsite in Tacoma, Washington area.

Right diagram is same as Fig 12 of reference (**6**) and based on tests with relative humidity apparatus No. 10 of 100 block in a special investigation of the rate of drying in storage at plant of Cleveland Builders Supply Co., Cleveland, Ohio.

The graph through the plotted points in the lower right diagram of Fig. 6 for block cured in high-pressure saturated steam at 350 to 365 F and made with four aggregates is of some interest. Although not too well defined by the scatter of points, it appears to indicate lower moisture content for a given relative humidity than block represented in the other diagrams of Fig. 6 which were cured in steam at atmospheric pressure at about 150 to 180 F. This can be expected from the results in Fig. 4 showing that the adsorbed water held by pastes cured at about 350 F, is appreciably less than for curing at 150 to 180 F.

The two diagrams of Fig. 7 also show a wide scatter of points when relative humidity is plotted against moisture content in per cent of total absorption as obtained in tests for moisture condition on separate halves of the same block. The results plotted in the right diagram are based on 100 comparable block made at the same plant on 10 days during a 13-day period. It will be apparent that if these block were dried so as to have a moisture content of either 20, 30, or 40 per cent of total absorption they would actually be in different degrees of dryness corresponding to a relative humidity range of about 25 to 33 per cent. The indicated range in actual dryness at a given percentage of total absorption is too wide to assure adequate control of shrinkage. On the other hand, note that if block are dried to a given relative humidity, say 70 per cent, some block may contain moisture equal to 20 per cent of absorption whereas others may contain 40 per cent of absorption, yet they have actually been dried to the same air-dry moisture condition and their actual shrinkage potential will have been minimized. Similar comments could be made for the results shown in the left diagram of Fig. 7 or for all diagrams in Fig. 6.

## CONCLUDING REMARKS

All the data cited in the preceding pages show that the current method for indicating state of dryness, based on per cent of total absorption, is fundamentally incorrect and must be practically ineffective in many cases. Besides being ineffective and incorrect, it offers no advantage with respect to speed and accuracy. Determining the per cent of total absorption requires from 48 to 72 hr, and it involves three separate weight measurements and computations, each subject to error.

Reliable drying shrinkage data indicate that it is possible for a block producer to comply with a specification which limits moisture content to 30 or 40 per cent of total absorption and yet the block will have the same or nearly the same potential for shrinking and cracking in the wall as block which have not been dried at all.

On the other hand, it is possible for a moisture limitation of 30 to 40 per cent of total absorption to be too severe and unrealistic, particularly with block made of dense heavyweight concrete having relatively low total absorption. The actual moisture content in an air-dry condition, of say 75 per cent relative humidity, may be normal but this would become a high value when expressed in per cent of a low total absorption value. This has forced some producers of quality block to reduce cement content and alter gradation of aggregate to produce a block of more porous concrete with greater absorption to enable them to comply more readily with a moisture content limitation based on total absorption. Obviously, a moisture content requirement which encourages increasing the water-absorptivity of block is undesirable and should be replaced.

These gross inconsistencies of the present ASTM method are contrary to the best interests of the concrete block producer and of the ultimate owner of the concrete masonry wall. They are completely eliminated by the Relative Humidity Method with which the block producer and consumer alike has a common, realistic basis for measuring and judging the moisture condition and potentiality for drying shrinkage of block to fit the requirements of any given locality of the country.

Although the cracking problem is very complex, it should be remembered that basically it is caused by excessive shrinkage movements and that there would be no problem if the material was dimensionally stable or strong enough to resist the tensile stresses caused by restrained shrinkage movements. Anything that is done to reduce shrinkage, from whatever cause, and to increase the tensile strength of the units will help to solve the cracking problem and usually at a lower cost than more control joints or other remedial measures.

*Acknowledgment:*

The author gratefully acknowledges the assistance of the following individuals whom he consulted during the preparation and review of this paper: Leo H. Corning, William Lerch, Treval C. Powers, George Verbeck, and Joseph J. Shideler, all of the Portland Cement Assn. He has also drawn freely on the information and concepts in papers by Ronald E. Copeland of the National Concrete Masonry Assn. and George W. Washa of the University of Wisconsin.

# APPENDIX I

## GENERAL CONSIDERATIONS OF THE CRACKING PROBLEM IN CONCRETE MASONRY WALLS[4]

Most cracks in concrete masonry walls could be eliminated by minimizing movements in the concrete masonry units themselves and by making the tensile strength of the units as high as is practicable. Movements in the units are not only the result of moisture and temperature changes but of chemical changes within the concrete when it absorbs carbon dioxide from the air. All three of these changes can cause shrinkage in the individual units and in the wall constructed of them. If the over-all shrinkage of the wall is restrained by foundation, floor or roof members, tensile stresses are produced which tend to produce cracks when the ultimate tensile strength of the concrete is exceeded. Although control joints, bond beams, and reinforcement in the mortar joints have been advocated and used, they are expensive means for minimizing the effect of movements which tend to take place in concrete masonry walls. Moreover, control joints are often unsightly, require maintenance and should be limited as far as practical. Hence, it is highly desirable to consider ways and means to increase tensile strength and to reduce wall movements by improving the dimensional stability of the units and by reducing their potential shrinkage by adequate drying.

The resistance of concrete to cracking is influenced by its tensile strength, its elongation under tensile stress at rupture, its tendency to shrink with temperature, moisture, and carbonation changes, and the degree of restraint imposed on it in service. The resistance to cracking, $R$, can be expressed by the ratio:

$$R = \frac{\text{unit tensile strength at rupture}}{\text{actual unit tensile stress}},$$

and can be expressed by the equation:

$$R = \frac{T}{S} = \frac{T}{(e_t + e_m + e_c)ED} \quad \ldots\ldots (1)$$

where:

$R$ = resistance to cracking

$T$ = tensile strength at rupture, psi.

$S$ = actual tensile stress imposed by restrained shrinkage, psi.

$e_t$ = unrestrained temperature shrinkage, in. per in.

$e_m$ = unrestrained moisture shrinkage, in. per in.

$e_c$ = unrestrained carbonation shrinkage, in. per in.

$E$ = secant modulus of elasticity as determined with the concrete subjected to sustained stress equal to 50 per cent of its ultimate strength, and

$D$ = degree of restraint, which is equal to the ratio:

$$D = \frac{\text{unit shrinkage prevented by restraint}}{\text{total unit unrestrained shrinkage}}$$

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[4] The author has used information and concepts on the cracking problem contained in two interesting papers, one by Saeman, Warren and Washa (**7**), the other by Copeland (**8**). These papers discuss various details and phases of this complex problem and are recommended for study. The various diagrams in reference (**8**) are of special interest.

Equation 1 for resistance to cracking, $R$, given herein is the same as the one on p. 850 of reference (**7**) in which $R$ was designated "relative immunity to cracking." Several of the symbols were also changed to be more descriptive of their meaning and the term, $e_c$, for carbonation shrinkage was added to the denominator of Eq 1. The symbol $D$ was used to represent degree of restraint instead of the symbol $I$, since this normally designates moment of inertia.

and can be expressed by the equation:

$$D = \frac{e_r}{(e_t + e_m + e_c)} = \frac{e_r}{e_r + e_y} = \frac{1}{1 + \dfrac{AE}{A_x E_x}} \quad (2)$$

where:

$e_r$ = unit shrinkage prevented by restraint
$e_y$ = unit movement of restraining members
$A$ = cross-sectional area of restrained concrete measured perpendicular to the direction of stress
$A_x$ = cross-sectional area of restraining members measured perpendicular to the direction of stress
$E_x$ = sustained modulus of elasticity of the restraining members

It will be apparent from Eq 1 that if $R = 1$, that is, if the actual tensile stress is equal to the ultimate tensile strength at rupture, a crack has occurred or is imminent. As $R$ becomes smaller than 1, the certainty of cracking increases, and as it becomes larger than 1, the probability of cracking decreases. When $e_r = 0$, $D = 0$, and there is no restraint. When $e_r = e_t + e_m + e_c$, $D = 1$, and there is complete restraint. However, for the usual case there is some yielding by the restraining member, designated by $e_y$.

The value of $A$ and of $A_xE_x$ and of $A/A_xE_x$ in Eq 2 varies over a wide range. With $A/A_xE_x$ equal to zero, $D$ equals 1, which indicates complete restraint. As the value of $A/A_xE_x$ increases, $D$ becomes smaller indicating a lesser degree of restraint.

Equations 1 and 2 can be used to estimate the effect of different variables as illustrated by Table XII of reference (**7**) and Table I of reference (**8**). For instance, it appears that resistance to cracking can be increased significantly by increasing the tensile strength $T$ of the concrete or by increasing the thickness of the face shells of the unit or by reducing the combined value of shrinkage $e_t + e_m + e_c$. An assumed improvement in one factor must be considered in its relation to other factors, as for instance if tensile strength is increased the effect on the ratio $T/E$ must be explored, etc.

The following paragraphs discuss what can be done about minimizing temperature, carbonation, and moisture changes.

*Temperature Changes.*—Changes in dimension with temperature changes depend mainly on the aggregate used in making the concrete unit. There is little that can be done about temperature movements, except perhaps selection of aggregate.

*Carbonation.*—The rate and ultimate amount of shrinkage of a concrete masonry unit with chemical changes in the cement paste, which occur with absorption of carbon dioxide from the air, is influenced by a number of factors such as the porosity of the cement paste and of the concrete, the moisture condition of the concrete, the method of curing, the presence of pozzolanic material, etc.

When the paste pores of a normally cured block are filled with water, such as at 100 per cent relative humidity, carbonation is insignificant but increases with drying as the pores empty and the paste becomes more susceptible to penetration by carbon dioxide until a maximum rate is attained at about 50 per cent relative humidity. As the humidity is lowered to 25 per cent, the paste does not contain enough water to facilitate carbonation and shrinkage again becomes insignificant. Roughly, the amount of shrinkage which tends to take place in a unit by carbonation is about equal to the drying shrinkage with moisture loss from a saturated condition to an air-dry condition. Although it would be desirable to stabilize units by a carbonation and drying treatment, there is no technical nor economical way thus far of rapidly carbonating them to remove carbonation shrinkage before they are placed in the wall.[5]

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[5] There may be four alternatives for minimizing the carbonation shrinkage: (1) dry block and expose them to air under covered storage for a long time, perhaps 1 yr; (2) dry block to a 50 per cent relative humidity condition and then expose them to artificial carbonation (and possibly simultaneous drying) in a treating chamber for at least 5 days; (3) produce block from a dense concrete and paste by a more favorable grading of aggregate and higher cement content than normal to retard the penetration by carbon dioxide; or (4) cure block in high pressure steam.

*Moisture Changes.*—Changes in dimension with moisture changes are influenced by a number of factors such as type of aggregate, the method of curing, and the moisture condition of the unit at the time it is placed in the wall in relation to its ultimate moisture condition in service.

Of the three factors which cause dimensional changes in the concrete masonry unit, that is, temperature, carbon dioxide, and moisture, only the latter can be minimized to a significant degree. This can be done by preshrinking the unit by adequate drying before it is placed in the wall. The degree of dryness will vary but for any given locality the concrete should be in an air dry condition corresponding to the average annual relative humidity of the outside air for that locality. This principle applies to all types of concrete masonry units regardless of type of aggregate or method of curing used.

It should be pointed out also that a marked reduction in shrinkage with moisture change (as much as 50 per cent reduction) can be attained by an adequate cycle of curing in high-pressure steam (autoclave curing) compared with moist curing at normal temperature or in steam at atmospheric pressure. Moreover, a quick pressure release at the end of the curing cycle results in a marked drying effect tending to bring concrete masonry units made with most aggregates except pumice to the desired degree of dryness.[6]

*Relative Humidity Method of Test:*

This test determines the moisture condition of hardened concrete in terms of relative humidity—the basic factor controlling the moisture condition of concrete exposed to the air. It is described in reference (1) and can be used in two ways:

(*a*) As an acceptance test that will indicate whether or not a given lot of block meet the degree of dryness required for a specific purpose.

(*b*) As a plant inspection test that will indicate to the block manufacturer the moisture condition of different lots of block at the plant so he will know when they are dry enough or what further drying is required for a specific purpose.

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[6] Recent tests at the Portland Cement Assn. Laboratories indicate that although autoclaved units undergo substantial carbonation shrinkage during exposure to high concentrations of carbon dioxide they do not develop significant shrinkage when exposed to carbon dioxide as normally found in the air. Moreover, these autoclaved units were at a moisture condition of 50 per cent relative humidity which represents a particularly favorable condition for carbonation in nonautoclaved units. Tests are under way to determine if autoclaved units are relatively immune to carbonation in air when they are at various moisture levels above 50 per cent relative humidity. If autoclaved cured units actually indicate a general immunity to carbonation shrinkage this would make autoclave curing still more desirable for the production of stable units and at no additional cost.

## REFERENCES

(1) Carl A. Menzel, "A Method for Determining the Moisture Condition of Hardened Concrete in Terms of Relative Humidity," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 1085 (1955); *Bulletin No. D4*, Research and Development Lab., Portland Cement Assn., Chicago, Ill.

(2) T. C. Powers and T. L. Brownyard, "Studies of the Physical Properties of Hardened Portland Cement Paste," *Proceedings*, Am. Concrete Inst., Vol. 43 (1947); *Bulletin No. 22*, Research and Development Lab., Portland Cement Assn., Chicago, Ill.

(3) George Verbeck, "Carbonation of Hydrated Portland Cement," Papers on Cement and Concrete, Am. Soc. Testing Mats. (1956). (Issued as separate publication *ASTM STP No. 205*.)

(4) N. C. Ludwig and S. A. Pence, "Properties of Portland Cement Pastes cured at Elevated Temperatures and Pressures," *Proceedings*, Am. Concrete Inst., Vol. 52, p. 673 (1956).

(5) Joseph J. Shideler, "Investigation of the Moisture-Volume Stability of Concrete Masonry Units," *Bulletin No. D3*, Research

and Development Lab., Portland Cement Assn., Chicago, Ill.

(6) Carl A. Menzel, "Report on Field Tests with Apparatus for Determining the Moisture Condition of Hardened Concrete Block by the Relative Humidity Method." Unpublished report on file at Portland Cement Assn., Chicago, Ill., Apr. 1955.

(7) J. C. Saemann, C. Warren and G. W. Washa, "Effect of Curing on the Properties Affecting Shrinkage Cracking of Concrete Block" *Proceedings*, Am. Concrete Inst., Vol. 51, p. 833 (1955).

(8) R. E. Copeland, "Shrinkage and Temperature Stresses in Masonry," *Proceedings*, Am. Concrete Inst, Vol. 53, p. 769 (1957).

# DISCUSSION

MR. R. E. COPELAND[1] (*presented in written form*).—For several years the engineering department as well as several member companies of the National Concrete Masonry Assn. have cooperated in developing data on the relative humidity test method. A series of tests at four plants was completed last January and reported to various interested individuals and organizations. Another series involving tests at eight plants is currently under way. In general we feel that the method has considerable merit but requires further evaluation over a broader range of conditions.

The obvious advantage of the method is that it can be performed fairly quickly with inexpensive, portable apparatus. It is not, however, always as fast a test as indicated by the author. In some of our tests as much as 24 hr was required to establish the equilibrium condition necessary for final readings of the relative humidity indicator.

The method offers other advantages although these appear to be of less importance. There are some apparent limitations. For example, our studies indicate it is a considerably less sensitive test in the higher than in the lower ranges of relative humidity.

There is little question but that the relative humidity method will become more widely used. In due course it undoubtedly will become an ASTM tentative method. Also, in time, it may replace the per cent of total absorption method in the standard specifications. This latter eventuality will be delayed, perhaps indefinitely, if the test method continues to be associated with unduly restrictive specification requirements.

The per cent of total absorption method is not nearly as bad as depicted in the paper. True, it could be improved by varying the allowable maximum values with the type of concrete and climatic conditions. However, in its present simple form as 40 per cent or 30 per cent it has served and is serving to direct attention to the need for specification control of the moisture condition of concrete units. Where a more exacting requirement is justified and will be enforced it can be incorporated in the specifications for the particular project or building.

Until recent years practically no attempt was made to enforce the moisture content limitation. Apparently, even large users and important specifiers of concrete masonry considered a moisture limitation a trivial detail of the ASTM Specifications. This situation is slowly changing. A substantial percentage of block is now subject to some degree of enforcement of the moisture limitation and an increasing number of producers are furnishing dry block as a matter of good business. There is evidence that this trend will continue, that an even wider and stricter enforcement of a reasonable but effective moisture limit will be supported by all groups concerned—industry, general interest, and user.

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[1] Director of Engineering, National Concrete Masonry Association, Chicago, Ill.

However, if the requirements are arbitrarily tightened to the point where they can neither be met nor enforced, the trend will be reversed. It is in this respect that the writer views the relative humidity method with certain misgivings, not so much because of any shortcomings in the method itself but because of the impossible moisture condition requirements being proposed in conjunction with it.

The author asserts that "...for any given locality the concrete should be in an air-dry condition corresponding to the average annual relative humidity of the outside air for that locality." Since, in most localities there are weeks and months when the average relative humidity substantially exceeds the annual average it should be evident to anyone that such a requirement cannot be met by any practicable means. In effect, the author is proposing that the block industry cease doing business during the fall, winter, and spring when the weather is notoriously damp and foggy in so many areas. Surely, that is not what he intended but the fact that a requirement so impossible of attainment and enforcement should be seriously proposed is reason enough for a cautious attitude toward the test method.

We know from field experience that block can be too dry for proper setting and bonding with mortar. Some retained moisture is necessary to control suction and prevent desiccating the mortar to where it has little strength and durability and develops separation cracks through which moisture readily penetrates. We do not know exactly what the critical moisture content is in this regard, but based on a few laboratory tests together with observations in the field, it appears to be not much less than 30 per cent of total absorption which corresponds roughly to the equilibrium moisture at 70 to 85 per cent relative humidity.

The problem of drying block must also be considered. Except during extremely favorable weather, the producer cannot depend on natural air drying, under cover, to attain a dryness under 30 per cent of absorption. It takes too long—from two to three months for hollow units and much more time for solid units. Many plants furnishing 30 per cent moisture content units have had to resort to forced drying in kilns using temperatures to about 200 F. While necessary to meet specifications, this practice is open to criticism since in some cases, not all, it sacrifices up to 35 per cent or more of the normal ultimate strength of the concrete. In the writer's view, specification requirements which necessitate drying processes damaging to the concrete will not serve the desired ends.

The only reason for specifying a dry block is to minimize drying shrinkage in the wall and so-called shrinkage cracking. It has no other purpose and improves no other property of the masonry units. On the contrary, the imposing of an excessively restrictive requirement as proposed by the author may, as pointed out, produce poorer instead of better masonry.

There are various ways to mitigate cracking in concrete masonry walls regardless of whether the cause is drying shrinkage, temperature shrinkage, or any of the other factors. These measures are rapidly becoming better known and more widely and effectively used. It is not technically sound to discount the progress in their use and take the position, as the author apparently has, that the only solution to the problem is to impose a very drastic dryness requirement.

It would be far better if the relative humidity test method could be considered strictly on its merits as a test method and not as a one-way street to a specification requirement which the block industry could not meet nor the using group enforce. If this could be done, considerable controversy and delay in acting on the test method could be avoided.

MR. CARL A. MENZEL (*author*).—I think Mr. Copeland has given an excellent discussion. The only points on which I differ with him are the practicability of reducing the moisture content to the expected relative humidity levels, either annual or monthly basis. It is not difficult to dry concrete block. Of course if you want to wait a long time to have it done by natural means in some times of the year, there is a considerable problem, and that time would be too long. But we have today accelerated means of drying units. Many concrete block manufacturers have these drying facilities with which block can be adequately dried within 9 or 10 hr. Hence it is not difficult for a progressive concrete block manufacturer to reduce the tendency to shrink and crack by adequate drying before the blocks go in the wall.

Mr. Copeland mentioned that often it takes 24 hr to get a final relative humidity value. I have pointed out that this may occur with block of nonuniform moisture content as when they are dry on the surface and obviously damp in the interior. Such block are not at equilibrium values with any given relative humidity. The test method outlines that readings of relative humidity should be continued until three successive readings, 5 min apart, are constant within $\frac{1}{2}$ per cent indicated relative humidity. Usually this end-point can easily be attained in 1 hr which is less than 1 per cent of the 72 hr that is now required by the current ASTM per cent of total absorption method. For all practical purposes this end-point provides a significant indication of the moisture condition of the concrete. No useful purpose is served by extending tests beyond 1 hr.

The drying of concrete block is not a difficult problem if it is tackled the way it deserves. You do not solve a problem by not doing anything about it but until recent years adequate drying was not required and only a few producers understood that it was desirable or necessary. Dryness was more or less accidental but inadequate drying has accentuated the drying shrinkage and cracking problem.

However, the time has come where the leaders in the industry have become cognizant of this problem. They know what to do about it. Many have put in dryers. Other producers have installed autoclaves in which they cure block in high pressure steam and obtain a rapid drying of the block during a rapid pressure release period. With autoclaving it is possible within 15 hr after molding to produce high quality block ready for delivery to the job in an adequately dry condition meeting the relative humidity test for any location in the country. In the case of block made with some of the very lightweight aggregates some additional drying will be required after autoclaving.

I think that everybody should give careful consideration to the need for drying and do something about it because we have a wonderful product in concrete masonry units. However, their tendency to shrink and crack should be minimized and I am absolutely confident this can be done if everybody does his fair share of the job.

We already know that with autoclave curing block producers have reduced the complaints about shrinkage cracking to insignificance. The relative humidity method has been developed to provide a more realistic method for determining moisture condition so that block will be preshrunk by adequate drying before they go in the wall. This should be of particular help to those producers who cannot afford the investment in autoclave curing. Hence, effective ways are available with which the concrete masonry industry can minimize wall cracking.

MR. C. C. CONNOR.[2]—This is a very informative paper. Preshrinking concrete blocks before laying would eliminate early shrinkage and generally give the mortar bonds time to develop strength before much moisture movement in the blocks would be likely to occur. Good mortar bonds would tend to restrain any subsequent moisture movement.

But it may be inferred from statements in the conclusions and the appendix that dimensional stabilization of the blocks by proper predrying would reduce their tendency to change volume in the wall when exposed to saturating rains or wide variations in humidity, and thus minimize cracking and limit the need for controlled joints. I doubt this is what the author meant.

My experience has been that cracking in concrete block walls frequently develops most seriously some months after construction. This would indicate a cause other than the initial condition of the blocks.

For clarification, I would like to ask the author if he believes that proper predrying of the blocks would provide dimensional stability which would minimize their moisture movement in the wall when exposed to rain and subsequent drying, or when exposed to wide changes in atmospheric humidity? And would predrying to a proper relative humidity greatly reduce the cracking in the blocks and at the mortar joints, and limit the need for control joints?

I would also like to ask the author if he feels that the usual practice of wetting concrete blocks slightly before laying should be discontinued for best results? Would this slight wetting upset the initial condition of relative humidity he advocates for curing, so that detrimental shrinkage would occur when the blocks dried out, or would the benefit to mortar bonds more than compensate?

MR. MENZEL.—I think we should endeavor to minimize these movements to which Mr. Connor refers, and if the blocks are dried adequately to begin with, you usually do not have too much swelling or too much shrinking. We should try to hit the intermediate point and the relative humidity method will indicate whether the blocks are initially at or near the right moisture condition. I know there will be variations in moisture content of individual block but when blocks are placed in the wall at or near the average relative humidity of the service condition wall cracking is reduced significantly.

Now in addition to the moisture shrinkage, there is the shrinkage which occurs when concrete absorbs carbon-dioxide from the air. Our laboratory is working very hard on means for reducing carbonation shrinkage. As stated by Mr. Copeland, control joints have been used in concrete masonry walls to reduce cracking and they serve a very useful purpose. Our idea is not to have too many control joints and reduce shrinkage as far as practical.

There are data developing which indicate that high-pressure steam curing will reduce the tendency for concrete block to absorb carbon dioxide from the air and to go through a further shrinkage. The results with high-pressure steam curing have indicated that we have very substantially reduced the tendency for cracking in walls constructed with autoclaved block.

MR. JOHN J. WHITE.[3]—Mr. Connor made a statement that has confused me no end. He speaks of the case of a block being placed in the wall and exposed to driving rain. If this were to a point where the block is completely saturated, the pore cells of the block may be completely filled with water and yet have no influence whatever on the moisture content. My understanding is that the cement paste alone is the ingredient of the block that is subject to either swelling or shrinkage. The fine and coarse aggregates, of course, are utterly inert and therefore can neither swell nor shrink within measurable limits.

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[2] Consulting Engineer, Masonry Construction and Maintenance, Verona, N. J.

[3] Chief Specifications Engineer, Parsons, Brinckerhoff, Hall and Macdonald, New York, N. Y.

My understanding is that the hydration of the cement causing the gel formation can be controlled and that the percentage of moisture is within the control of that gel formation and that the ultimate soaking of a block and the filling of the pore cells has utterly no bearing whatever on moisture content.

I should like to have some elucidation on that.

MR. CONNOR.—I think it is generally conceded that concrete blocks will expand when thoroughly wetted, and that they will shrink when dried. Saxer and Toennies[4] tabulated the shrinkage that occurred in various kinds of concrete blocks when dried by different methods after several hours immersion in water. Those concrete blocks were dried at 73 F and 50 per cent relative humidity for 10 days preceding the tests. These blocks should have been dimensionally stabilized by this predrying, but it did not prevent volume changes when they were wetted and then dried.

MR. COPELAND.—The effect of wetting and drying of the masonry wall after it has been constructed is less, so far as cracking development is concerned, than that of laying a wet block into the wall. The reason for this is that the same restraints that cause shrinkage stresses also restrain the tendency of the masonry to expand with wetting.

I was very much interested in what Mr. Connor said, and I would like to ask Mr. Connor whether his organization ever test block for their moisture condition? As I indicated in my discussion, very few organizations pay any attention to the moisture content requirement of block and I think that is one of the reasons why we have had trouble which could have been prevented had specifiers and users read the specifications and taken steps to see that the block at least met the moisture content requirements indicated.

Mr. Connor, does your organization take block from the job, test them for moisture condition? If they do not meet the requirement, do you reject the block and require block that do meet the specification?

MR. CONNOR.—It has not been the practice in the company I was formerly associated with to test the moisture content of concrete blocks before use. It was only required that they be thoroughly dried and be protected from rain during the period between delivery and laying in the wall. Testing for moisture content might have improved results in some cases.

The main question I have raised with the author of this paper is whether or not proper predrying would greatly reduce the moisture movement of the blocks in the wall when they are wetted by long-continued rains and then dry out, or when they are subjected to wide variations in atmospheric humidity.

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[4] E. L. Saxer and H. T. Toennies, "Measuring Shrinkage of Concrete Block," see p. 988, this publication.

# FREEZING-AND-THAWING RESISTANCE OF CONCRETE AS AFFECTED BY THE METHOD OF TEST*

By H. L. Flack[1]

## Synopsis

The data presented in this paper are a compilation of test results of several investigations performed by the Bureau of Reclamation to determine the factors which may influence results of laboratory freezing-and-thawing tests. Several combinations of freezing-and-thawing periods including four tentative ASTM methods were used to test the effect of initial cures, degree of saturation, and specimen size and shape. In addition, the time-temperature relationships established for different locations in an 18-in. cylindrical specimen to simulate the relative effect on structural concrete are shown for various patterns of freezing and thawing.

The main purpose of this paper is to ascertain the influence of factors which might in part explain the lack of agreement between results of tests which utilized different laboratory freezing and thawing cycles and in some instances possible reasons for the poor correlation existing between the laboratory indicated durability and the service records of field structures. Data from the standard Bureau of Reclamation test (1)[2] and the four tentative ASTM methods[3] to measure the resistance of non-air- and air-entrained concretes to freezing-and-thawing actions are evaluated, as well as several different initial cures and subsequent treatments, degrees of saturation, and size and shape of specimens.

Except where otherwise noted, the air-entrained concretes used in these investigations contained a good sound natural aggregate graded up to $\frac{3}{4}$-in. maximum size and approximately 3 to 5 per cent air. Non-air-entrained concretes contained the same aggregates and entrapped air from 1.4 to 2.3 per cent. Extreme care was exercised in

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[1] Materials Engineer, Bureau of Reclamation, Denver, Colo.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1095.

[3] Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 3, p. 192.

Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 3, p. 200.

Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 3, p. 196.

Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 3, p. 204.

## TABLE I.—EFFECT OF DIFFERENT INITIAL CURING PERIODS ON THE RESISTANCE OF CONCRETE TO FREEZING-AND-THAWING ACTION.[a]

MIX DATA FOR AIR-ENTRAINED CONCRETE.

| Mix | Cement Content, lb | Water Content, lb | Water: Cement Ratio, W/C | Sand, per cent | Slump, in. | Air Content, per cent |
|---|---|---|---|---|---|---|
| 1A...... | 589 | 300 | 0.51 | 45 | 3.0 | 4.2 |
| 2A...... | 591 | 301 | 0.51 | 45 | 2.7 | 4.0 |
| 3A...... | 588 | 300 | 0.51 | 45 | 2.5 | 4.8 |
| 4A...... | 540 | 297 | 0.55 | 42 | 2.6 | 3.2 |
| 5A...... | 504 | 278 | 0.55 | 42 | 2.8 | 5.1 |

MIX DATA FOR PLAIN CONCRETE.[b]

| Mix | Cement Content, lb | Water Content, lb | Water: Cement Ratio, W/C | Sand, per cent | Slump, in. | Air Content, per cent |
|---|---|---|---|---|---|---|
| 1........ | 660 | 337 | 0.51 | 49 | 3.3 | 1.8 |
| 2........ | 663 | 338 | 0.51 | 49 | 3.3 | 1.4 |
| 3........ | 649 | 331 | 0.51 | 50 | 2.5 | 2.3 |

MATERIALS USED

| | |
|---|---|
| Cement .......... | Laboratory blend, type II |
| Air-entraining Agent .......... | Vinsol resin |
| Aggregate .......... | Sand—Clear Creek; Specific gravity 2.67; Abs 0.6<br>Gravel—Clear Creek; Specific gravity 2.65; Abs 0.5 |
| Method of Test .... | Test No. 1 (USBR) (1) |

| Mix | Curing of Specimens Prior to Freezing-and-Thawing Test | | | | Age of Specimen at Start of Freezing-and-Thawing Test, days | Air-Entrained Concrete—3- by 6-in. Specimens | | | | | Plain Concrete[b]—3- by 6-in. Specimens | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Initial Curing Period, days, Fog at 73 F | Second Curing Period | | | | Number of Cycles and Expansion | | | | Number of Cycles to 25 per cent Weight Loss | Number of Cycles and Expansion | | | | Number of Cycles to 25 per cent Weight Loss |
| | | | | | | To Obtain 5 per cent Weight Loss | | To Obtain 12 per cent Weight Loss | | | To Obtain 5 per cent Weight Loss | | To Obtain 12 per cent Weight Loss | | |
| | | Time, days | Average Temperature, deg Fahr | Condition | | Cycles | Expansion, per cent | Cycles | Expansion, per cent | | Cycles | Expansion, per cent | Cycles | Expansion, per cent | |
| 1A and 1..... | 14 | .. | .. | .. | 14 | 470 | 0.27 | 627 | .. | 660 | .. | .. | .. | .. | 50 |
| | 28 | .. | .. | .. | 28 | 345 | 0.10 | 760 | 0.32 | 770 | .. | .. | .. | .. | 130 |
| | 14 | 76 | 73 | 50 % RH | 90 | 785 | 0.00 | 1705 | 0.01 | 2860 | 80 | 0.03 | 95 | 0.05 | 150 |
| 2A and 2..... | 14 | .. | .. | .. | 14 | 345 | 0.16 | 485 | 0.60 | 675 | .. | .. | .. | .. | 50 |
| | 28 | .. | .. | .. | 28 | 345 | 0.09 | 438 | 0.20 | 660 | 50 | 0.62 | 100 | .. | 140 |
| | 14 | 76 | 73 | 50 % RH | 90 | 1090 | 0.02 | 1950 | 0.02 | 2870 | 115 | 0.04 | 150 | .. | 200 |
| 3A and 3..... | 28 | .. | .. | .. | 28 | 310 | 0.12 | 513 | 0.55 | 600 | 217 | 0.57 | .. | .. | 280 |
| | 90 | .. | .. | .. | 90 | 565 | 0.08 | 825 | 0.23 | 1090 | 320 | 0.36 | .. | .. | 450 |
| | 16 | 164 | 73 | 50 % RH | 180 | 1025 | 0.04 | 1848 | 0.02 | 2580 | 216 | 0.50 | 262 | 0.71 | 320 |
| | 7 | .. | .. | .. | 7 | 248 | 0.09 | 418 | 0.46 | 550 | .. | .. | .. | .. | 65 |
| | 14 | .. | .. | .. | 14 | 310 | 0.18 | 475 | 0.58 | 500 | .. | .. | .. | .. | 160 |
| | 28 | .. | .. | .. | 28 | 330 | 0.14 | 528 | 0.55 | 620 | 302 | 0.76 | .. | .. | 360 |
| | 63 | .. | .. | .. | 63 | 462 | 0.07 | 690 | 0.35 | 820 | 392 | 0.61 | 452 | .. | 520 |
| | 119 | .. | .. | .. | 119 | 460 | 0.05 | 765 | 0.09 | 1125 | 565 | 0.56 | 714 | .. | 790 |
| | 180 | .. | .. | .. | 180 | 545 | 0.06 | 850 | 0.13 | 1100 | 540 | 0.42 | 732 | 0.75 | 820 |

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3A and 3 | 7 | 7 | 73 | 50% RH | 14 | 755 | 0.09 | 1092 | 0.38 | 1350 | 199 | .. | .. | .. | 225 |
| | 7 | 21 | 73 | 50% RH | 28 | 740 | 0.07 | 1280 | 0.20 | 1640 | 174 | 0.05 | 223 | .. | 260 |
| | 7 | 56 | 73 | 50% RH | 63 | 588 | 0.02 | 1468 | 0.04 | 2730 | 225 | 0.02 | 338 | 0.10 | 480 |
| | 7 | 112 | 73 | 50% RH | 119 | 650 | 0.02 | 1688 | 0.03 | 3630 | 178 | 0.08 | 285 | .. | 460 |
| | 7 | 173 | 73 | 50% RH | 180 | 415 | 0.02 | 1408 | 0.02 | 3790 | 176 | 0.06 | 286 | .. | 500 |
| | 14 | 7 | 73 | 50% RH | 21 | 640 | 0.21 | 928 | 0.11 | 1320 | .. | .. | .. | .. | 200 |
| | 14 | 21 | 73 | 50% RH | 35 | 802 | 0.09 | 1250 | 0.23 | 1745 | .. | .. | .. | .. | 310 |
| | 14 | 49 | 73 | 50% RH | 63 | 1090 | 0.04 | 1900 | 0.07 | 2785 | 338 | 0.04 | 485 | .. | 530 |
| | 14 | 105 | 73 | 50% RH | 119 | 830 | 0.03 | 2340 | 0.05 | 3560 | 271 | 0.04 | 433 | 0.13 | 650 |
| | 14 | 166 | 73 | 50% RH | 180 | 560 | 0.01 | 1695 | 0.03 | 3640 | 190 | 0.04 | 323 | 0.29 | 440 |
| | 28 | 7 | 73 | 50% RH | 35 | 553 | 0.08 | 878 | 0.21 | 1130 | 189 | 0.45 | .. | .. | 310 |
| | 28 | 35 | 73 | 50% RH | 63 | 1128 | 0.06 | 1925 | 0.07 | 2670 | 325 | 0.24 | 435 | 0.54 | 470 |
| | 28 | 91 | 73 | 50% RH | 119 | 1425 | 0.04 | 2313 | 0.05 | 3480 | 214 | 0.29 | 267 | .. | 350 |
| | 28 | 154 | 73 | 50% RH | 180 | 1800 | 0.03 | 1832 | 0.04 | 3110 | 212 | 0.47 | 231 | .. | 260 |
| 4A | .. | 28 | 73 | Sealed | 28 | 375 | .. | 775 | .. | 940 | .. | .. | .. | .. | .. |
| | .. | 90 | 73 | Sealed | 90 | 300 | .. | 695 | .. | 1917 | .. | .. | .. | .. | .. |
| | .. | 180 | 73 | Sealed | 180 | 296 | .. | 650 | .. | 1455 | .. | .. | .. | .. | .. |
| 5A | | 28 | 50 | Sealed | 28 | 450 | | 550 | | 720 | .. | .. | .. | .. | .. |
| | | 90 | 50 | Sealed | 90 | 380 | | 770 | | 1520 | .. | .. | .. | .. | .. |
| | | 180 | 50 | Sealed | 180 | 360 | .. | 780 | .. | 1460 | .. | .. | .. | .. | .. |

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[a] Resistance to freezing and thawing as determined by standard Bureau of Reclamation freezing-and-thawing test.

[b] Concrete without entrained air.

the batching, mixing, and fabrication of all concretes, utilizing, where applicable, standard ASTM and Bureau of Reclamation procedures.

Briefly, the five methods of test employed consisted of two different rapid cycles of freezing and thawing in water, a rapid cycle of freezing in air and thawing in water, and two slow cycles, one of which employed freezing in air and one freezing in water, and thawing in water in both. Important features of each test are tabulated below:

| Test | Test Designation | Freezing | | Thawing | | Length of Cycle |
|---|---|---|---|---|---|---|
| | | In | At | In | At | |
| No. 1..... | USBR[a] | Water | 10 F | Water | 70 F | 1½ hr freezing<br>1½ hr thawing |
| No. 2..... | ASTM C 290[b] | Water | 0 F | Water | 40 F | 1 hr freezing<br>1 hr thawing |
| No. 3..... | ASTM C 292[b] | Water | 0 F | Water | 70 F | 24 hr freezing<br>24 hr thawing |
| No. 4..... | ASTM C 291[b] | Air | 0 F | Water | 40 F | 80 min freezing<br>40 min thawing |
| No. 5..... | ASTM C 310[b] | Air | 0 F | Water | 40 F | 17 hr freezing<br>7 hr thawing |

[a] Reference (**1**).
[b] See footnote 3.

To evaluate results of tests, various criteria are employed and will be described where used.

## DISCUSSION OF TEST RESULTS—INITIAL CURING

Of importance in the evaluation of test data derived from the various freezing-and-thawing tests, and also the distress, caused by freezing action in field structures, are the conditions to which the concretes are subjected throughout the periods of initial curing, subsequent exposure, and testing. Since continuous water or fog curing has proven beneficial to many of the properties of concrete, all methods to the present time require initial periods of moist curing. Figure 1, which is a plot of data contained in Table I, shows the effects on freezing and thawing resistance of three different cures: continuous fog, sealed at two temperature conditions, and a combination of fog plus periods of drying under controlled conditions.

Since similar trends were noted for both types of concrete, only test results of air-entrained concretes are graphically presented in Fig. 1. In this series the USBR test (No. 1) (1) was used. Curve 1 shows that the resistance of air-entrained concrete increases with increasing periods of fog curing; and, within the limits of this investigation, it increased from 550 cycles after 7 days to double that figure after 180 days of fog curing.

Specimens continuously sealed at both 50 F and 73 F, curves 2 and 3, respectively, did not follow the same trend developed by their fog-cured companions discussed above. At 28 days, resistance to freezing and thawing developed by the fog and both sealed cures were approximately the same. However, from this point on freezing and thawing resistance was greatly benefited by further sealed curing at both temperatures. Of the two temperatures used, the higher one, 73 F, proved to be most beneficial. However, both temperatures produced optimum resistance at 90 days.

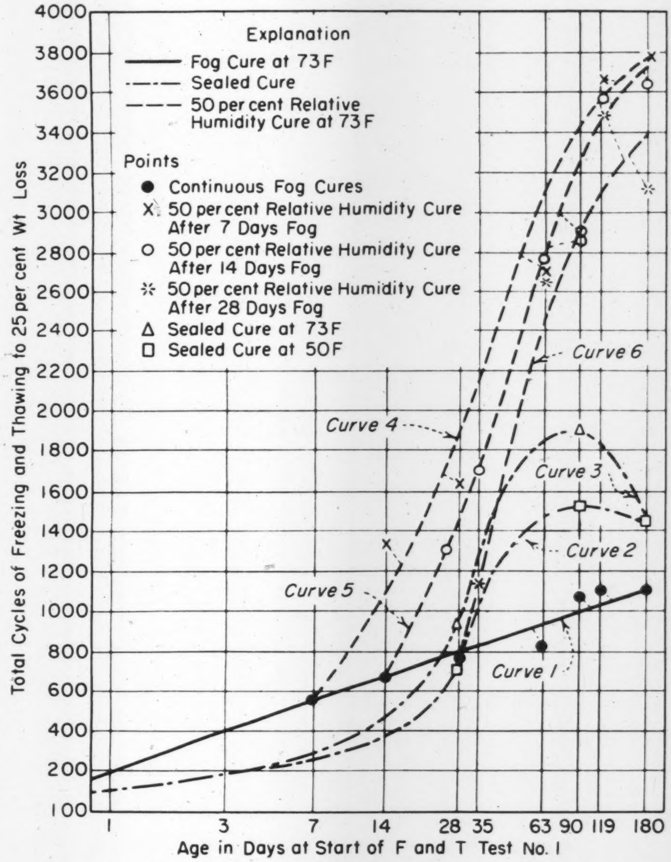


FIG. 1.—The Conditions Under Which Specimens Are Initially Cured Greatly Influence the Ultimate Resistance to Freezing and Thawing Developed by Air-Entrained Concrete.

The three curing combinations in which the concrete was first fog cured for 7, 14, and 28 days, and subsequently stored at 50 per cent relative humidity (curves *4*, *5*, and *6*) proved to be even more beneficial to the resistance of the concrete than was the sealed cure, with failures averaging 3500 cycles as compared with failures in the neighborhood of 1460 cycles for the sealed cure and 1100 cycles for the continuously fog-cured concretes at the age of 180 days. There also is some evidence that freezing-and-thawing resistance is not improved by fog curing in excess of 7 days where subsequent drying at 50 per cent relative humidity is employed.

In the curing of concrete, the humidity tends to equalize with the humidity of the medium surrounding it (2,3). This change in humidity is, of course, caused by the transfer of water from the condition of high humidity to that of low humidity. Since the hydration process of the cement tends to reduce the humidity within the concrete from an initial value of 100 per cent (4), conditions can exist such that transfer of water vapors can be inward as well as outward, and this transfer is accelerated or retarded by temperature changes. For fog curing, the direction of flow is inward, and, as this process continues, the air pores and capillaries are being filled with water until finally a relatively high degree of saturation is reached which is conducive to relatively rapid deterioration.

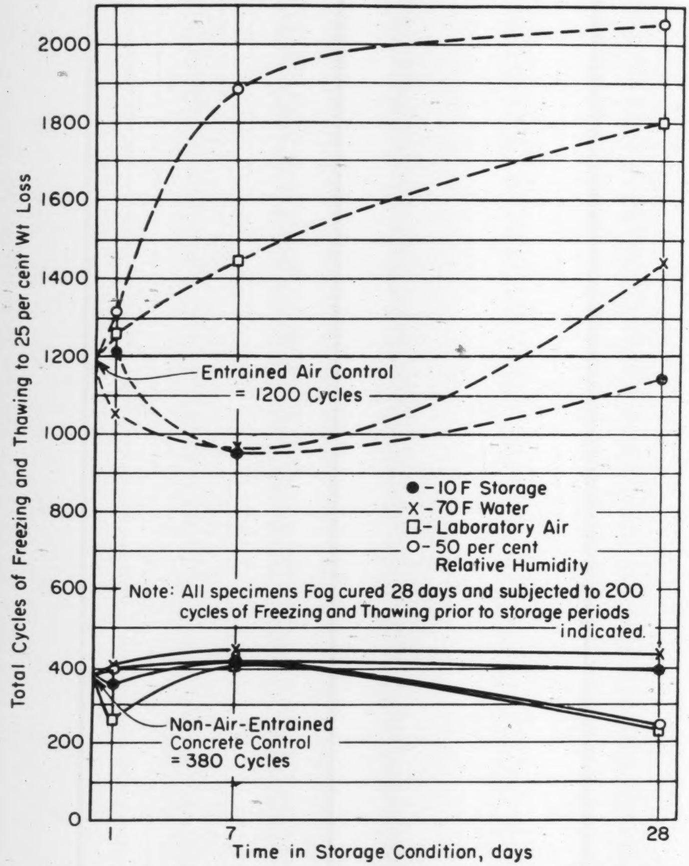


FIG. 2.—Beneficial Effects of Periodic Dry and 50 per cent Relative Humidity Cures Especially on Air-Entrained Concrete on Resistance to Freezing and Thawing Illustrate the Benefits on Field Structures by Intermittent and Seasonal Changes in Weather Conditions.

In the case of sealed cured concrete, there is neither gain nor loss of moisture prior to freezing-and-thawing test, and in the combination fog plus dry cure, there is an outward transfer of moisture. Both of these cures then reduce, in varying degree, the amount of freezable water in the specimen at the start of the test and render the concretes thus cured more resistant to freezing and thawing than companion concrete fog cured; the greater the water loss the greater the resistance developed.

Concrete in field structures also receives various types of initial curing as well as intermittent dry-and-wet curing conditions caused by spasmodic and seasonal changes in temperatures and humidity. The short periods under dry conditions will no doubt produce beneficial effects on the durability of concrete by the removal of some or all the free water from the air voids and capillaries in the concrete. However, prolonged periods of extremely dry conditions may retard the hydration rate of the cement which may cause cracks from autogenous and drying shrinkage which together will provide a ready means for penetration of water and water vapors into the concrete during periods of relatively high humidity and moisture.

### SUBSEQUENT CURING AND EXPOSURE

The effect of different types of intermittent curing on the durability of concrete is illustrated in Fig. 2 plotted from test data of another investigation (**5**) in which non-air- and air-entrained specimens were subjected to different periods of various exposures after the completion of 200 cycles of freezing and thawing. Only relatively small differences caused by different curing conditions were noted in the non-air-entrained concrete; however, the air-entrained concrete was significantly affected by the type and length of the exposure period. For all storage conditions in which a degree of drying, with its attendant loss of moisture, was achieved, the resistance to freezing and thawing progressively improved with increased curing time under these conditions of exposure up to 28 days. Such was not the case where soaking in water at 70 F or freezing at 10 F was used. Only after 28 days in water was the concrete as durable as the control concrete.

In general, the ability of concrete, both in the form of laboratory specimens and in field structures, to withstand the disruptive forces of freezing depends to a large extent on the amount of water in the air voids and capillaries of the concrete at the time of freezing. The amount in which this water will exist in turn depends on the age of the concrete as well as the different curing conditions and subsequent treatment to which the concrete has been exposed.

### DEGREE OF SATURATION

As mentioned in the preceding paragraphs, the initial fog curing periods combined with periods of controlled drying at 50 per cent relative humidity are very beneficial to the freezing-and-thawing resistance of air-entrained concrete. The additional water required to maintain hydration at the maximum rate (**4**) under the given conditions of test is provided during these initial fog curing periods. During subsequent treatments at lower humidity, the actual amount of uncombined or freezable water will gradually decrease due to continued hydration of the cement and the losses due to evaporation.

In the freezing-and-thawing procedures in which the specimens are submerged in water, the specimens progressively become more saturated depending on the number, size, and spacing of the air voids as well as the water tightness of the mortar layer at the surface of the concrete. To compare the rate at which both plain and air-entrained concretes approached saturation, forty 3 by 6-in specimens fabricated from two mixes, using white cement and a good quality limestone, were fog cured for 7 days. The specimens were then placed in watertight tubes, 4 cylinders to each tube, submerged in a 0.5 per cent solution of phenolphthalein, and the tubes were placed in the USBR (test No. 1) (**1**) freezing-and-thawing test apparatus. As the dye solution penetrates the concrete, the point of maximum penetration is

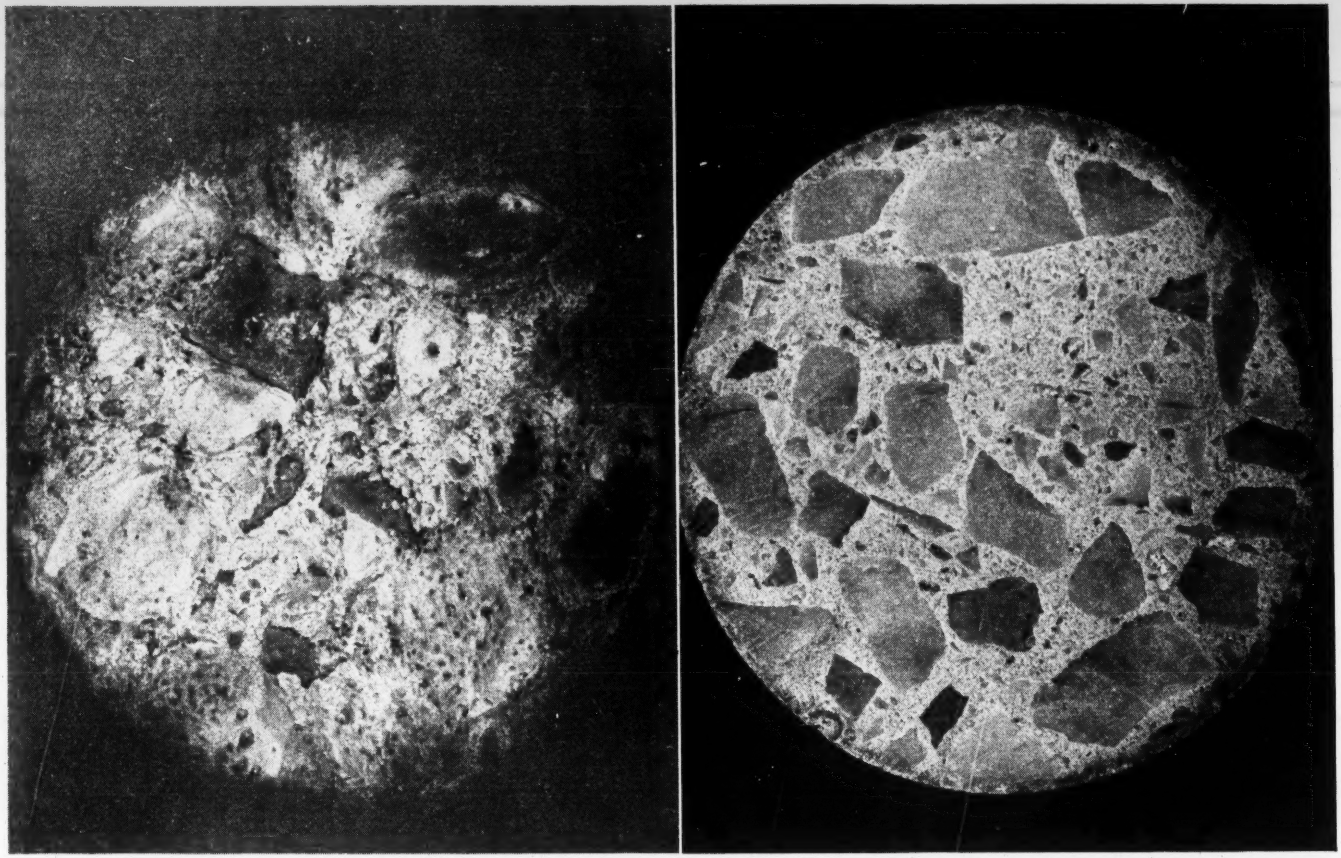


FIG. 3.—The Penetration of the 0.5 per cent Phenolphthalein Solution as Indicated by the Darkened appearance of Sections of the Freshly Broken Surface of Plain Concrete (left view) and the Narrow Peripheral Ring on the Cut Surface of the Air-Entrained Concrete (right view). Both Concretes Had Been Subjected to 20 Cycles of Freezing and Thawing.

shown by the width of the pinkish colored ring on a cut or broken surface of the concrete (Fig. 3).

Unfortunately, the plain concrete specimens deteriorated at a very fast rate and failure of these specimens occurred at 20 cycles, at which time they were initially observed. On the other hand, the air-entrained concrete showed no distress at 20 cycles and but little distress after 200 cycles. The rapid failure of the non-air-entrained concrete is attributed to two factors: (1) the absence of an adequate air void system, and (2) the relatively rapid approach to the point of critical saturation (6). Figure 3 shows the amount of penetration at 20 cycles for both non-air- and air-entrained concrete. The phenolphthalein solution, as indicated by the darkened surfaces, penetrated a considerable distance into the non-air-entrained concrete, especially around the larger aggregate particles. This penetration was facilitated by the cracks which are readily visible in this figure. The penetration of the dye solution into the air-entrained concrete, on the other hand, was very small at 20 cycles, as shown by the narrow ring of less than $\frac{1}{8}$ in. near the surface. As the test on the air-entrained concrete specimens continued, the width of this ring has slowly increased, but has not at the end of 200 cycles reached the point of penetration of that of the plain concrete at 20 cycles. Other studies in this investigation of penetration of water into the concrete through formed and troweled surfaces has shown that penetration of the top troweled surface of a specimen is much more rapid than that of the formed surface. This phenomenon is probably due to formation of capillaries to the top surface during the bleeding of the fresh concrete.

## SPECIMEN SIZE

In the testing program performed in cooperation with Committee B-1 of the Highway Research Board, concrete bars, 3 by 3 by 16 in., were tested to evaluate the freezing-and-thawing resistance of three types of air-entrained concrete using the four different ASTM tentative methods of test.[3] Three concretes of different potential resistance to freezing and thawing were used. Two of the concretes contained well-rounded saturated fine and coarse aggregate with both low (2.75 ± 0.25 per cent) and high (7.0 ± 0.5 per cent) designed air contents. The third concrete contained the same natural fine aggregate used in the above-mentioned concretes and a soft, saturated, crushed clay-bearing limestone coarse aggregate. The designed air content of this concrete was 6.5 to 7.5 per cent. To determine the effect of specimen size and shape, 3 by 6-in. cylindrical specimens were also fabricated from each mix and, with the exception of test No. 5, were tested in each of the ASTM methods with their companions. In order to compare the behavior of the various concretes when subjected to the ASTM methods, Nos. 2 to 5, and the USBR method, No. 1, the cylinders fabricated for test No. 5 were subjected to test No. 1 conditions. Based on increments of 10, 25, and 40 per cent reduction in sonic modulus of elasticity, the average results from periodic observations of all specimens for (1) the number of cycles, (2) expansion, and (3) weight change in the four ASTM tests and test No. 1 have been summarized in Table II.

In a statistical analysis of individual test results, coefficients of variation of the number of cycles at the failure point of 40 per cent reduction in sonic modulus specified by ASTM were computed to compare the uniformity of test results obtained through the use of bars or cylinders, and the effect of the different methods on results obtained. The coefficients are tabulated under the heading "V%" in Fig. 4 outside the charts

## TABLE II.—EFFECT OF DIFFERENT METHODS OF TESTING ON THE RESISTANCE TO FREEZING AND THAWING OF CONCRETE BARS AND CYLINDERS FABRICATED FROM MIXES USING TWO TYPES OF COARSE AGGREGATE.

### MIX DATA.

| Mix | Cement Content, lb | Water Content, lb | Water: Cement Ratio, W/C | Sand, per cent | Slump, in. | Air Content, per cent | Mix | Cement Content, lb | Water Content, lb | Water: Cement Ratio, W/C | Sand, per cent | Slump, in. | Air Content, per cent | Mix | Cement Content, lb | Water Content, lb | Water: Cement Ratio, W/C | Sand, per cent | Slump, in. | Air Content, per cent |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. 1L | 536 | 257 | 0.48 | 45.3 | 3.0 | 2.8 | No. 1A | 546 | 238 | 0.435 | 42.9 | 3.0 | 6.4 | No. 1B | 531 | 257 | 0.484 | 44.3 | 3.2 | 6.8 |
| No. 2L | 540 | 258 | 0.48 | 45.3 | 3.7 | 2.8 | No. 2A | 546 | 238 | 0.435 | 42.9 | 3.7 | 6.4 | No. 2B | 528 | 256 | 0.485 | 44.3 | 3.5 | 7.5 |
| No. 3L | 538 | 257 | 0.48 | 45.3 | 3.8 | 2.6 | No. 3A | 542 | 236 | 0.435 | 42.9 | 3.2 | 6.9 | No. 3B | 530 | 257 | 0.485 | 44.3 | 3.9 | 7.3 |
| No. 4L | 533 | 255 | 0.48 | 45.3 | 2.9 | 3.0 | No. 4A | 547 | 238 | 0.435 | 42.9 | 3.1 | 6.3 | No. 4B | 545 | 261 | 0.485 | 44.3 | 2.1 | 6.8 |

### FREEZING-AND-THAWING TEST RESULTS

| Mix | Age of Fog Cured Specimen at Start of Freezing-and-Thawing Test, days | Method of Freezing-and-Thawing Test | | | | | | | Average Freezing-and-Thawing Test Results on 3 by 6-in. Cylinders | | | | | | | | | Average Freezing-and-Thawing Test Results on 3 by 3 by 16-in. bars | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Subjected to Freezing | | | Subjected to Thawing | | | Number of Cycles per Week | Number of Cycles, Expansion, and Weight Loss at | | | | | | | | Durability Factor[b] | Number of Cycles, Expansion, and Weight Loss at | | | | | | | | | Durability Factor[b] |
| | | | | | | | | | 10 per cent Loss in E[a] | | | 25 per cent Loss in E[a] | | | 40 per cent Loss in E[a] | | | 10 per cent Loss in E[a] | | | 25 per cent Loss in E[a] | | | 40 per cent Loss in E[a] | | | |
| | | Time, hr | Average Temperature, deg Fahr | In Presence of | Time, hr | Average Temperature, deg Fahr | In Presence of | | Cycles | Expansion, per cent | Weight Loss, per cent[d] | Cycles | Expansion, per cent | Weight Loss, per cent[d] | Cycles | Weight Loss, per cent[d] | | Cycles | Expansion, per cent | Weight Loss, per cent[d] | Cycles | Expansion, per cent | Weight Loss, per cent[d] | Cycles | Expansion, per cent | Weight Loss, per cent[d] | |
| CONCRETE USING A HARD WELL-ROUNDED GRAVEL WITH 2.5 TO 3.0 PER CENT AIR | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 1L | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 18 | 0.002 | 0.1 | 56 | 0.045 | 1.5 | 68 | .. | 13.6 | 12 | 0.003 | −0.01 | 32 | 0.017 | −0.01 | 43 | 0.020 | 0.02 | 7.1 |
| | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 20 | 0.002 | 0.8 | .. | .. | .. | 43 | .. | 14.6 | 15 | 0.013 | 0.03 | 23 | 0.019 | 0.15 | 30 | 0.030 | 0.18 | 6.1 |
| | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 13 | 0.008 | 0.5 | 31 | 0.045 | 1.2 | 82 | .. | 11.4 | 11 | 0.005 | 0.03 | 26 | 0.015 | 0.05 | 33 | 0.036 | 0.06 | 6.7 |
| Average | | | | | | | | | 17 | 0.004 | 0.5 | 44 | 0.045 | 1.4 | 64 | | 13.2 | 13 | 0.007 | 0.02 | 27 | 0.017 | 0.06 | 35 | 0.029 | 0.09 | 6.6 |
| 2L | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 10 | 0.029 | 0.1 | 22 | 0.057 | 3.1 | 49 | 7.4 | 9.8 | 46 | 0.039 | 0.53 | 67 | 0.101 | 1.43 | 79 | 0.124 | 1.78 | 15.8 |
| | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 53 | 0.037 | 0.8 | 74 | 0.083 | 2.5 | 85 | 4.2 | 17.0 | 40 | 0.031 | 0.41 | 53 | 0.052 | 0.70 | 66 | 0.094 | 1.28 | 13.3 |
| | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 21 | 0.021 | 0.3 | 51 | 0.072 | 1.1 | 59 | 2.2 | 11.8 | 74 | 0.042 | 0.87 | 96 | 0.072 | 1.16 | 111 | 0.094 | 1.79 | 22.1 |
| Average | | | | | | | | | 28 | 0.029 | 0.4 | 49 | 0.071 | 2,2 | 64 | 4.6 | 12.9 | 53 | 0.037 | 0.60 | 72 | 0.075 | 1.10 | 85 | 0.104 | 1.62 | 17.1 |
| 3L | 14 | 1⅓ | 0 | Air | ⅔ | 40 | Water | 84 | 13 | 0.017 | 0.1 | 34 | 0.046 | 0.2 | 73 | 0.4 | 14.6 | 11 | 0.007 | 0.05 | 94 | 0.039 | 0.27 | 131 | 0.079 | 0.21 | 26.3 |
| | 14 | 1⅓ | 0 | Air | ⅔ | 40 | Water | 84 | 23 | 0.006 | .. | 55 | 0.041 | .. | 73 | .. | 14.6 | 24 | 0.095 | 0.23 | 86 | 0.046 | 0.25 | 121 | 0.085 | 0.13 | 24.3 |
| | 14 | 1⅓ | 0 | Air | ⅔ | 40 | Water | 84 | 10 | 0.030 | .. | 38 | 0.137 | .. | 57 | .. | 11.4 | 30 | 0.015 | 0.02 | 73 | 0.033 | 0.07 | 102 | 0.078 | 0.05 | 20.1 |
| Average | | | | | | | | | 15 | 0.018 | 0.1 | 42 | 0.075 | 0.2 | 68 | 0.4 | 13.5 | 22 | 0.039 | 0.10 | 84 | 0.039 | 0.20 | 118 | 0.081 | 0.13 | 23.6 |
| 4L | 14 | [c] | | | | | | | 7.8 | 0.035 | 0.04 | 19 | 0.087 | 0.11 | 31 | 0.16 | 6.2 | Test discontinued after 400 cycles | | | | | | | | | 101.2 |
| | 14 | | | | | | | | 9.2 | 0.014 | 0.02 | 23 | 0.036 | 0.05 | 37 | 0.08 | 7.3 | | | | | | | | | | 101.7 |
| | 14 | | | | | | | | 6.5 | 0.029 | 0.03 | 16 | 0.072 | 0.06 | 26 | 0.10 | 5.2 | | | | | | | | | | 102.9 |
| Average | | | | | | | | | 7.8 | 0.026 | 0.03 | 19 | 0.065 | 0.07 | 31 | 0.11 | 6.2 | | | | | | | | | | 101.9 |

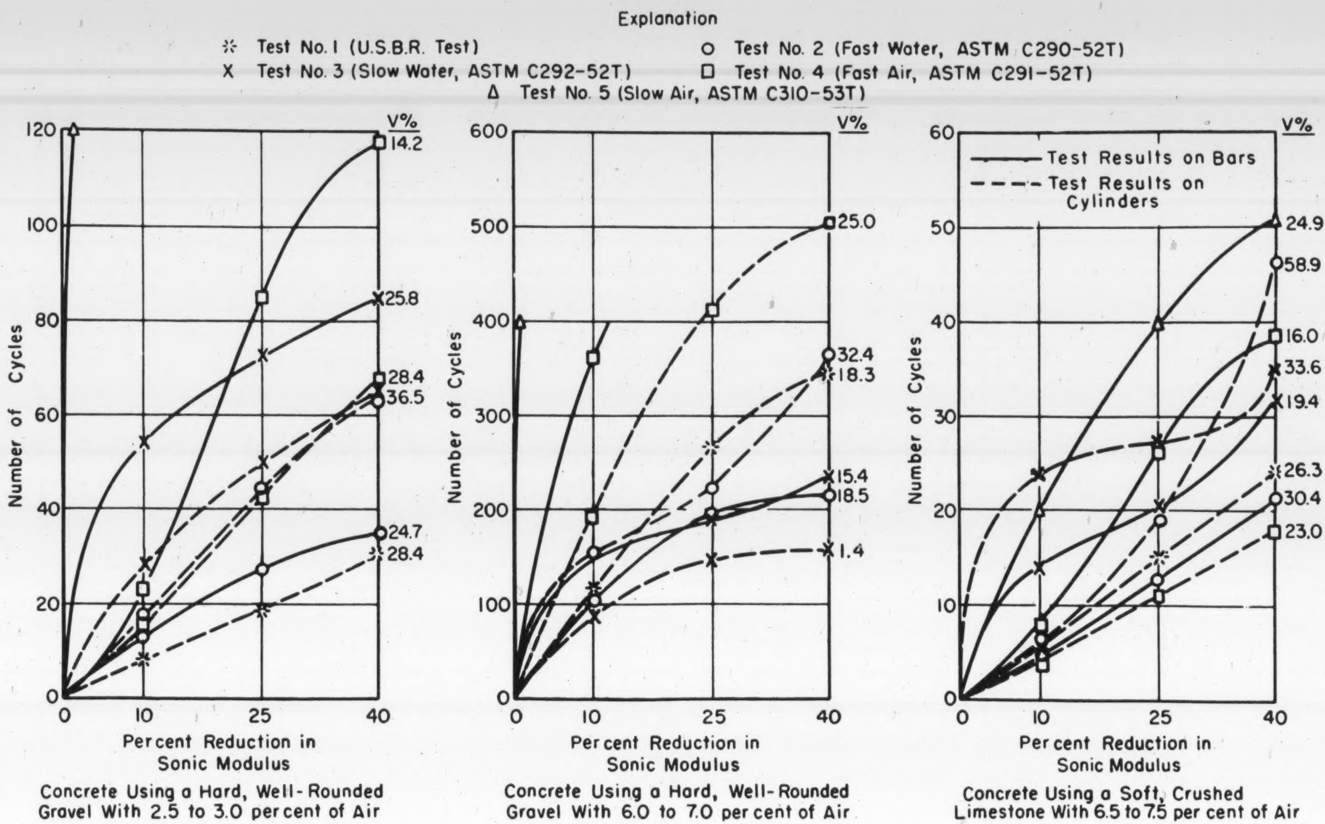
| | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| CONCRETE USING A HARD WELL-ROUNDED GRAVEL WITH 6.5 TO 7.0 PER CENT AIR | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 1A | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 79 | 0.015 | 0.3 | 186 | 0.040 | 2.2 | 281 | 6.3 | 56.1 | 97 | 0.007 | 1.09 | 218 | 0.022 | 2.66 | 234 | 0.026 | 3.08 | 46.9 |
| | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 244 | 0.052 | 2.1 | ... | ... | ... | 460 | 9.8 | 83.5 | 76 | 0.008 | 0.83 | 139 | 0.021 | 1.56 | 160 | 0.034 | 1.88 | 32.0 |
| | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 126 | 0.017 | 0.2 | 264 | 0.049 | 5.8 | 356 | 14.4 | 68.5 | 141 | 0.009 | 1.38 | 222 | 0.021 | 2.45 | 245 | 0.032 | 2.88 | 48.9 |
| Average | | | | | | | | | 150 | 0.028 | 0.9 | 225 | 0.045 | 4.0 | 366 | 10.2 | 69.4 | 105 | 0.008 | 1.10 | 193 | 0.021 | 2.22 | 213 | 0.031 | 2.61 | 42.6 |
| 2A | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 102 | 0.026 | 2.5 | 146 | 0.051 | 3.1 | 157 | 3.4 | 30.4 | 159 | 0.044 | 1.88 | 201 | 0.059 | 3.30 | 246 | 0.083 | 4.60 | 49.6 |
| | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 78 | 0.012 | 1.1 | 146 | 0.022 | 5.0 | 157 | 7.8 | 31.4 | 123 | 0.026 | 1.63 | 166 | 0.035 | 2.23 | 225 | 0.064 | 4.32 | 44.9 |
| | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 74 | 0.025 | 0.8 | 147 | 0.054 | 3.4 | 156 | 3.9 | 31.2 | 145 | 0.033 | 1.14 | 194 | 0.053 | 3.88 | 236 | 0.069 | 5.18 | 45.6 |
| Average | | | | | | | | | 85 | 0.021 | 1.5 | 146 | 0.042 | 3.8 | 157 | 5.0 | 31.0 | 142 | 0.034 | 1.55 | 187 | 0.049 | 3.14 | 236 | 0.072 | 4.70 | 46.7 |
| 3A | 14 | 1 1/3 | 0 | Air | 2/3 | 40 | Water | 84 | 154 | 0.014 | 0.1 | 484 | 0.040 | 1.1 | 598 | 0.1 | 83.0 | Test discontinued after 400 cycles—No failures | | | | | | | | | 96.1 |
| | 14 | 1 1/3 | 0 | Air | 2/3 | 40 | Water | 84 | 148 | 0.004 | 0.2 | 471 | 0.051 | 0.4 | 589 | 0.4 | 80.8 | | | | | | | | | | 95.8 |
| | 14 | 1 1/3 | 0 | Air | 2/3 | 40 | Water | 84 | 270 | 0.042 | 0.4 | 289 | 0.051 | 0.4 | 324 | 0.1 | 78.7 | | | | | | | | | | 84.7 |
| Average | | | | | | | | | 191 | 0.020 | 0.2 | 415 | 0.047 | 0.6 | 504 | 0.2 | 80.8 | | | | | | | | | | 92.2 |
| 4A | 14 | [c] | | | | | | | 90 | 0.030 | 1.8 | 226 | 0.048 | 3.4 | 304 | 6.2 | 60.7 | Test discontinued after 400 cycles—No failures | | | | | | | | | 107.2 |
| | 14 | | | | | | | | 28 | 0.012 | 0.1 | 226 | 0.024 | 3.6 | 343 | 7.4 | 68.6 | | | | | | | | | | 105.4 |
| | 14 | | | | | | | | 220 | 0.043 | 2.6 | 342 | 0.046 | 5.4 | 396 | 7.4 | 79.1 | | | | | | | | | | 108.9 |
| Average | | | | | | | | | 113 | 0.028 | 1.5 | 265 | 0.039 | 4.1 | 348 | 7.0 | 69.5 | | | | | | | | | | 106.9 |
| CONCRETE USING A SOFT, CRUSHED LIMESTONE WITH 6.5 TO 7.5 PER CENT AIR | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 1B | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 11.0 | 0.031 | −0.06 | 30 | 0.081 | −0.25 | 81 | 0.5 | 16.1 | 4.5 | 0.010 | 0.13 | 10 | 0.029 | 0.23 | 16 | 0.060 | 0.31 | 3.7 |
| | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 4.3 | 0.015 | −0.07 | 11 | 0.040 | −0.17 | 24 | 0.0 | 4.7 | 4.0 | 0.008 | 0.06 | 12 | 0.036 | 0.19 | 18 | 0.070 | 0.28 | 4.3 |
| | 14 | 1 | 0 | Water | 1 | 40 | Water | 84 | 4.2 | 0.001 | −0.08 | 17 | 0.042 | −0.26 | 33 | −0.5 | 6.7 | 5.3 | 0.013 | 0.12 | 18 | 0.031 | 0.27 | 30 | 0.063 | 0.42 | 6.1 |
| Average | | | | | | | | | 6.5 | 0.016 | −0.07 | 19 | 0.054 | −0.23 | 46 | 0.0 | 9.2 | 4.6 | 0.010 | 0.10 | 13 | 0.032 | 0.23 | 21 | 0.064 | 0.34 | 4.7 |
| 2B | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 7.1 | 0.017 | 0.23 | 15 | ... | 0.51 | 21 | 0.7 | 4.2 | 15 | 0.041 | −0.01 | 24 | 0.084 | −0.01 | 35 | 0.137 | 0.01 | 7.1 |
| | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 39 | 0.021 | 0.39 | 27 | 0.049 | 2.07 | 33 | 5.3 | 6.6 | 13 | 0.040 | −0.07 | 17 | 0.071 | −0.06 | 26 | 0.129 | 0.0 | 5.2 |
| | 14 | 24 | 0 | Water | 24 | 73 | Water | 3.5 | 26 | 0.044 | 0.24 | 38 | 0.110 | 0.17 | 52 | 0.2 | 10.2 | 13 | 0.040 | −0.06 | 20 | 0.075 | −0.07 | 34 | 0.146 | −0.01 | 6.9 |
| Average | | | | | | | | | 24 | 0.027 | 0.29 | 27 | 0.080 | 0.92 | 35 | 2.1 | 7.0 | 14 | 0.040 | −0.05 | 20 | 0.077 | −0.05 | 32 | 0.137 | 0.0 | 6.4 |
| 3B | 14 | 1 1/3 | 0 | Air | 2/3 | 40 | Water | 84 | 4.3 | 0.027 | 0.05 | 11 | 0.069 | 0.13 | 17 | 0.20 | 34.4 | 11 | 0.017 | 0.0 | 29 | 0.050 | −0.05 | 43 | 0.122 | −0.40 | 8.6 |
| | 14 | 1 1/3 | 0 | Air | 2/3 | 40 | Water | 84 | 5.9 | 0.037 | 0.01 | 15 | 0.094 | 0.02 | 24 | 0.02 | 47.6 | 7.1 | 0.020 | −0.02 | 20 | 0.054 | −0.16 | 33 | 0.116 | −0.32 | 6.6 |
| | 14 | 1 1/3 | 0 | Air | 2/3 | 40 | Water | 84 | 3.2 | 0.030 | 0.04 | 8 | 0.074 | 0.10 | 13 | 0.16 | 25.6 | 7.4 | 0.020 | −0.01 | 28 | 0.061 | −0.28 | 38 | 0.108 | −0.42 | 7.4 |
| Average | | | | | | | | | 4.5 | 0.031 | 0.03 | 11 | 0.079 | 0.08 | 18 | 0.13 | 35.9 | 8.5 | 0.019 | −0.01 | 26 | 0.055 | −0.16 | 38 | 0.115 | −0.38 | 7.5 |
| 4B | 14 | [c] | | | | | | | 7.9 | 0.035 | 0.03 | 20 | 0.087 | 0.08 | 32 | 0.12 | 6.3 | 24 | 0.025 | 0.15 | 48 | 0.061 | 0.00 | 71 | 0.099 | −0.10 | 14.2 |
| | 14 | | | | | | | | 5.7 | 0.031 | 0.06 | 14 | 0.077 | 0.14 | 23 | 0.22 | 4.6 | 22 | 0.019 | 0.18 | 39 | 0.061 | 0.02 | 49 | 0.087 | −0.47 | 34.7 |
| | 14 | | | | | | | | 4.4 | 0.031 | 0.02 | 11 | 0.077 | 0.05 | 17 | 0.07 | 3.5 | 14 | 0.021 | −0.14 | 32 | 0.058 | −0.22 | 34 | 0.097 | −0.30 | 8.8 |
| Average | | | | | | | | | 6.0 | 0.032 | 0.04 | 15 | 0.080 | 0.09 | 24 | 0.14 | 4.8 | 20 | 0.022 | 0.06 | 40 | 0.060 | −0.07 | 51 | 0.094 | −0.29 | 19.2 |

[a] Dynamic modulus of elasticity as determined by standard procedure in accordance with ASTM Method C 215.[4]

[b] Durability factor based on 40 per cent reduction in dynamic modulus of elasticity and 300 cycles for termination of test.

[c] Bar specimens, 3 by 3 by 16-in., were subjected to a freezing period in air at 0 F for 17 hr and followed by thawing in water at 40 F for 7 hr, and the companion cylinders were tested in the standard Bureau of Reclamation test which consists of $1\frac{1}{2}$ hr freezing at 10 F and $1\frac{1}{2}$ hr thawing at 70 F.

[d] Negative values indicate weight gain instead of weight loss.



Concrete Using a Hard, Well-Rounded Gravel With 2.5 to 3.0 percent of Air

Concrete Using a Hard, Well-Rounded Gravel With 6.0 to 7.0 per cent of Air

Concrete Using a Soft, Crushed Limestone With 6.5 to 7.5 per cent of Air

FIG. 4.—Relationships of Number of Cycles with Reduction of 10, 25, and 40 per cent in Sonic Modulus for Both Bars and Cylinders for the Three Types of Concretes.

and opposite the curves representing each type of concrete and specimen. Examination of these values indicates that the coefficients of variation of both bars and cylinders varied widely, from 14.2 to 30.4 and 1.4 to 58.9, respectively.

Further analysis shows that in the three methods of test (tests Nos. 2, 3, and 4) in which both bars and cylinders were tested, the indicated durability of the bars varied somewhat less than that of the cylinders, the coefficients of variation being 22.7 and 32.2 per cent, respectively. The indicated durability of the bars was not consistently greater or less than that of the cylinders, but both were significantly affected by the type of freezing (in water or in air) to which they were subjected. For the specimens subjected to the fast water method, the bars were less durable than cylinders. The reason for this is believed to be due to the higher percentage of troweled surface in the bars which, as discussed under "degree of saturation," provide a relatively greater number of capillaries which facilitate the entrance of water.

### DIFFERENT METHOD OF TESTING

The relative behaviors of the three concretes as measured by number of cycles are plotted in the three graphs in Fig. 4. This figure shows that the different methods of test produced a wide range of durabilities as measured by the number of cycles to 40 per cent reduction in sonic modulus for all three types of concrete, but did evaluate the different types of concretes in the same relative order. The order of increased severity of test methods, as shown by the test data on bars, is (1) the fast water, (2) slow water, (3) fast air, and (4) the slow air. No like order was established where cylindrical specimens were used.

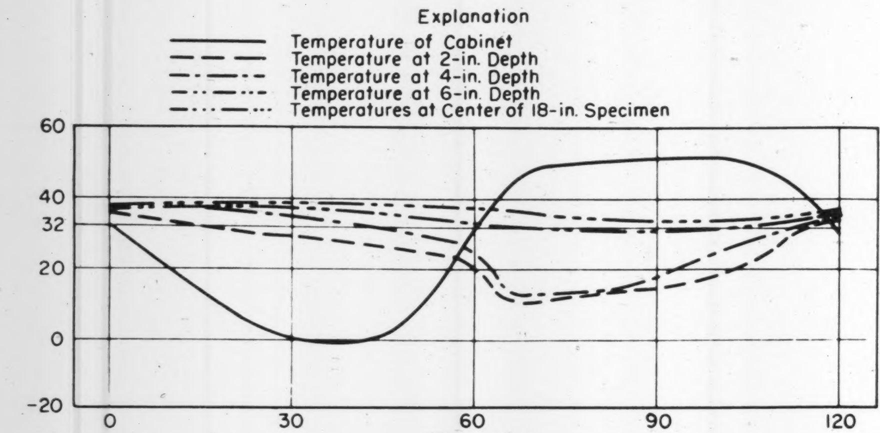
From these data, it may be seen that the primary factor influencing the indicated durability as far as the test is concerned was the media, water or air, in which the concrete was frozen and not the rates of freezing employed in the tests. The temperature differential between the freezing and thawing portions of the cycle and duration of the cycle appeared to have only minor effects on the reduction in sonic modulus, length change, and weight change (Table II).

From the standpoint of the different methods of test, the coefficients of variation given in Fig. 4 indicate that test No. 3 (slow water) employing a 48-hr cycle with equal periods of freezing at 0 F and thawing at 70 F produced results of less variance than any of the other tests.

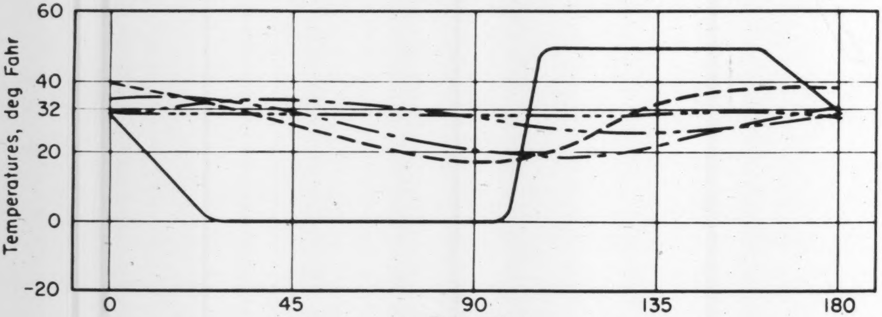
In spite of the wide variations in the freezing-and-thawing resistance indicated by the various methods of test, and in any particular test by the three concretes which varied in quality because of low air content or poor aggregate, the freezing-and-thawing method of test, regardless of cycle used, is considered to be a good test for the over-all quality of the concrete. Of the three concretes discussed in this section, one contained good quality materials and adequate air for the maximum size of aggregate used and developed relatively good resistance to freezing and thawing. However, when the quality of this concrete was lowered, either by the use of a poor quality coarse aggregate or by the entrainment of insufficient air, the resistance was drastically reduced. Thus it is emphasized that, in the matter of freezing-and-thawing resistance, there is no substitute for either quality in materials or the entrainment of sufficient air to provide a good air-void system with small spacing factor.

### TIME-TEMPERATURE RELATIONSHIPS IN A LARGE SPECIMEN

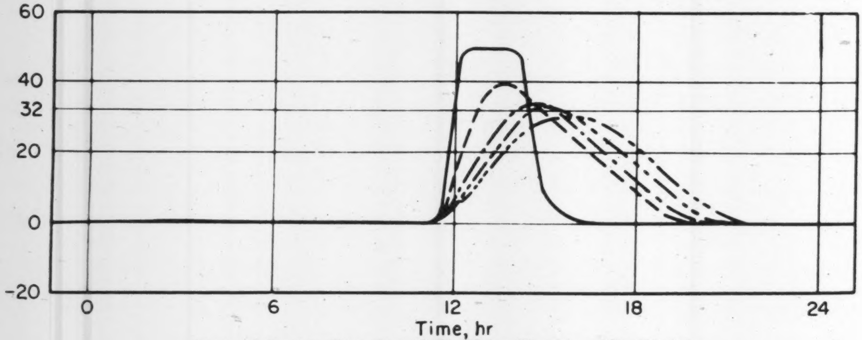
At the present time, a series of tests is being conducted on large 18-in. by 18-in. cylindrical air-entrained speci-



Type of Cycle: Freezing (0 F) 1 hr Thawing (50 F) 1 hr



Type of Cycle: Freezing (0 F) 100 min Thawing (50 F) 80 min



Type of Cycle: Freezing (0 F) 22 hr Thawing (50 F) 2 hr

FIG. 5.—Recorded Temperatures at Different Locations in an 18-in. Cylindrical Specimen Throughout Three Different Freezing-and-Thawing Cycles.

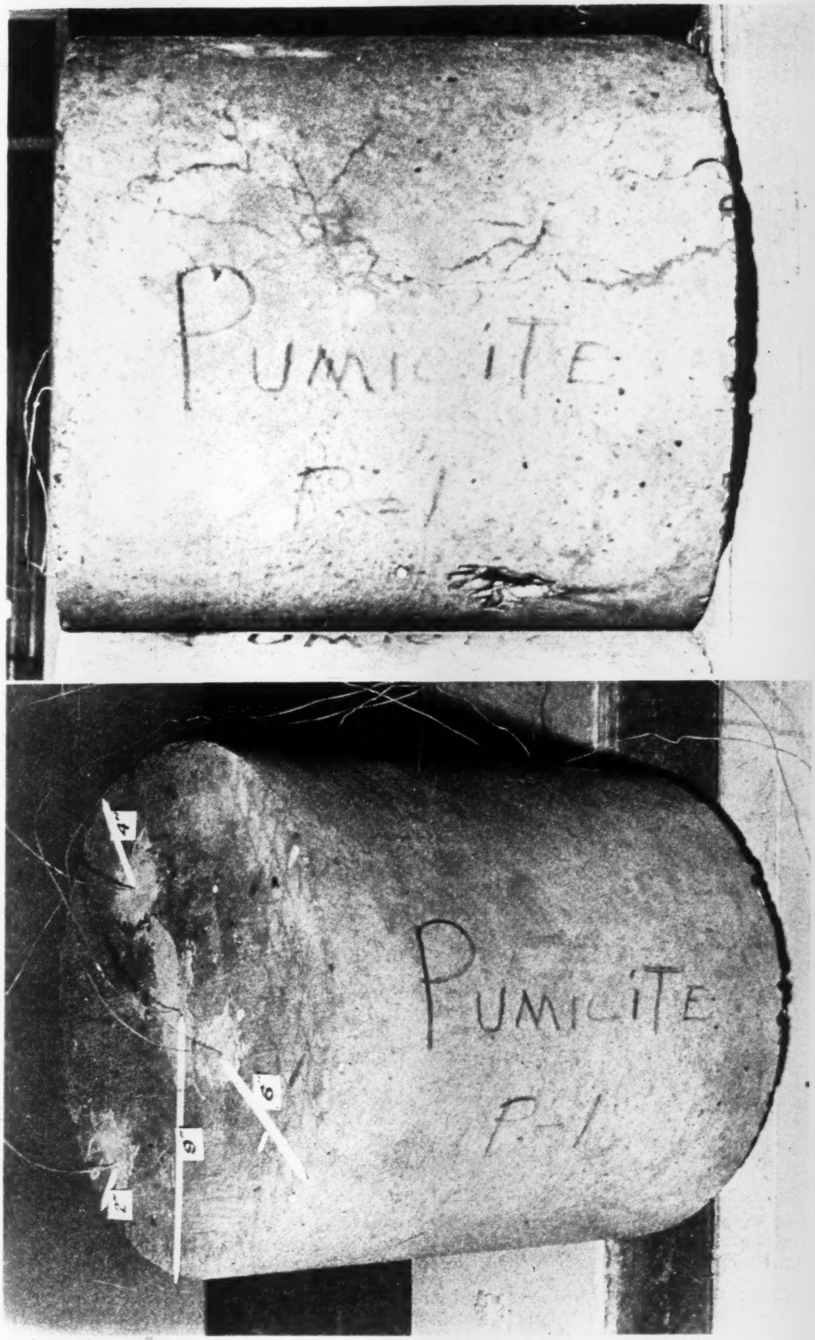

PUMICITE
P-1
PUMICITE
P-1
4"
6"
9"
2"

FIG. 6.—Appearance of 18-in. Cylindrical Specimen Complete with Thermocouples at the Beginning of Test (left view) and After 400 Cycles (right view) of Freezing and Thawing.

Explanation: Area of Concrete With Temperatures Above 32 F is Represented by the Hatched Area on the Chart

Area of Concrete With Temperatures Below 32 F is Represented by the Clear Area on the Chart

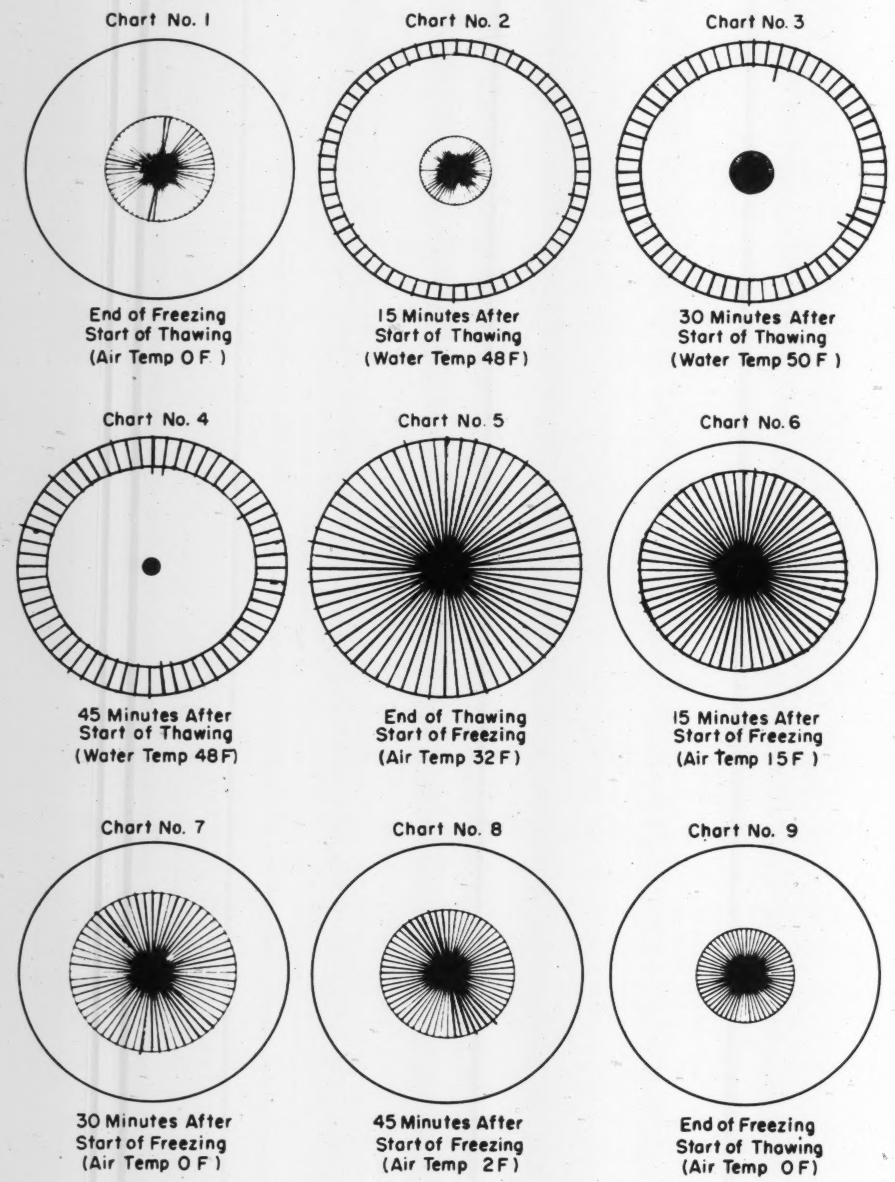


Chart No. 1
Chart No. 2
Chart No. 3
End of Freezing
Start of Thawing
(Air Temp 0 F )
15 Minutes After
Start of Thawing
(Water Temp 48 F)
30 Minutes After
Start of Thawing
(Water Temp 50 F )
Chart No. 4
Chart No. 5
Chart No. 6
45 Minutes After
Start of Thawing
(Water Temp 48 F)
End of Thawing
Start of Freezing
(Air Temp 32 F)
15 Minutes After
Start of Freezing
(Air Temp 15 F )
Chart No. 7
Chart No. 8
Chart No. 9
30 Minutes After
Start of Freezing
(Air Temp 0 F )
45 Minutes After
Start of Freezing
(Air Temp 2 F)
End of Freezing
Start of Thawing
(Air Temp 0 F)

FIG. 7.—Charts Representing Cross-Sectional Areas of an 18-in. Cylindrical Specimen for Temperatures Above and Below 32 F at 15-min Intervals Throughout a 2-hr Cycle After Stable Conditions Were Reached.

Explanation: Area of Concrete With Temperature Above 32 F is Represented by the Hatched Area on the Chart

Area of Concrete With Temperature Below 32 F is Represented by the Clear Area on the Chart

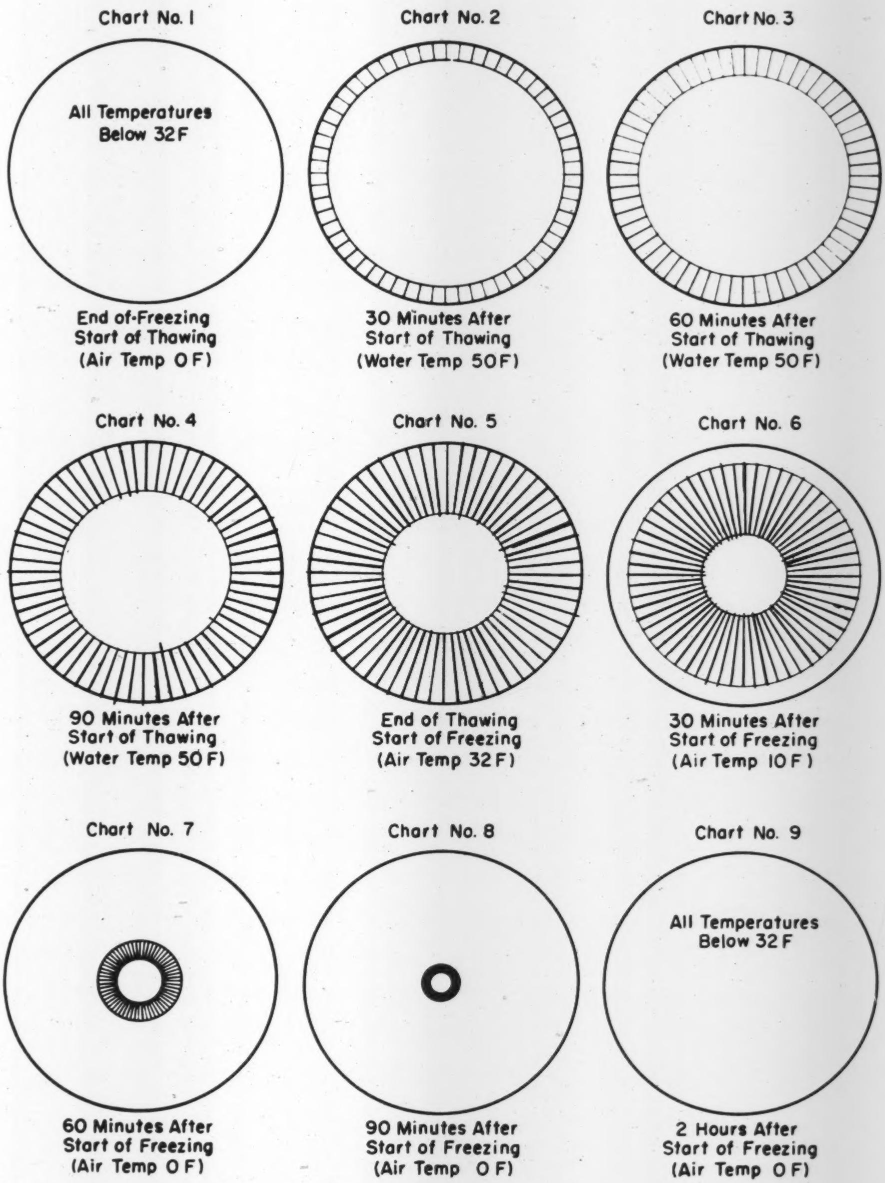


Chart No. 1
All Temperatures Below 32 F
End of-Freezing Start of Thawing (Air Temp 0 F)
Chart No. 2
30 Minutes After Start of Thawing (Water Temp 50 F)
Chart No. 3
60 Minutes After Start of Thawing (Water Temp 50 F)
Chart No. 4
90 Minutes After Start of Thawing (Water Temp 50 F)
Chart No. 5
End of Thawing Start of Freezing (Air Temp 32 F)
Chart No. 6
30 Minutes After Start of Freezing (Air Temp 10 F)
Chart No. 7
60 Minutes After Start of Freezing (Air Temp 0 F)
Chart No. 8
90 Minutes After Start of Freezing (Air Temp 0 F)
Chart No. 9
All Temperatures Below 32 F
2 Hours After Start of Freezing (Air Temp 0 F)

FIG. 8.—Charts Representing Cross-Sectional Areas of an 18-in. Cylindrical Specimen for Temperatures Above and Below 32 F at 30-min Intervals Throughout 2-hr Thawing Portion and the First 2 hr of the Freezing Portion of a 24-hr Cycle.

mens containing aggregate graded up to 6-in. maximum size. The purpose of this test is to study the time-temperature relationship at various points in the specimen and the deteriorative effect when large specimens are subjected to different temperatures for different periods of freezing and thawing, and to simulate the action to which field structures are subjected during adverse climatic conditions such as long freezing conditions with intermittent short periods of thawing.

Figure 5 shows three typical curves of the temperatures recorded by thermocouples placed as shown in Fig. 6 at locations of 2, 4, 6, and 9 in. from the vertical outside surface of the 18-in. specimen. These thermocouples were embedded to a depth of 9 in., midpoint in the height of the specimen. Cabinet temperatures were controlled at 0 F and 50 F during the freezing-and-thawing cycles, respectively. From the 2-hr cycles curves shown at the top of Fig. 5, it is noted that the rate of temperature reduction at different depths during the freezing cycle gradually decreases, and for this particular cycle, only the outer portion of concrete to a depth of approximately 6 in. is subjected to freezing temperatures. In contrast, when the freezing-and-thawing cycle is changed to 100 min below and 80 min above 32 F respectively (middle curve) the temperature at the center of the specimen approaches 32 F and only minute changes in this temperature were recorded during subsequent cycles of either freezing or thawing.

The bottom curve, Fig. 5, shows results of tests which simulate conditions to which field structures are often subjected during the cold season. Temperatures were recorded during a 24-hr cycle of which only 2 hr were utilized for the thawing period at 50 F. Under these conditions, the temperatures at 2, 4, 6, and 9 in. from the vertical surface reach maximum temperatures of 40, 34, 33, and 30 F, respectively. In this case, the center portion from the depth of approximately $8\frac{1}{2}$ in. to the center did not reach temperatures above 32 F throughout the entire cycle.

In Figs. 7 and 8, charts representing cross-sectional areas of the 18-in. cylindrical specimen for temperatures above and below 32 F at different time intervals are shown for the 2-hr and 24-hr cycles. In Fig. 7, it may be seen that the large specimen contains alternate zones of frozen and thawed concrete, and at no time is completely frozen, whereas for the 24-hr cycle, Fig. 8, the center portion of the concrete remains at temperatures below 32 F throughout the thawing phase of the cycle and the thawing action continues to penetrate toward the center as the cabinet temperatures are reduced below freezing. In the freezing phase, an outer zone of frozen concrete is formed advancing toward the center of the specimen. This phenomenon creates a confined volume of thawed concrete which gradually diminishes as it progresses toward the center of the specimen. The width of this confined volume (thawed concrete) and the rate at which it progresses toward the center depends on the temperatures of the freezing phase of the cycle, time duration, and thermal properties of the concrete. As the concrete becomes saturated due to absorption of water during thawing phase, the decreasing volume of the thawed concrete will reach a point at which insufficient space is available for the expansion of the absorbed water, and consequently, cracking and deterioration will occur. The cracked condition of the large specimen, shown to the right in Fig. 6, is attributed to this type of failure.

## CONCLUSIONS

1. Freezing-and-thawing resistance of fog-cured air-entrained concrete increases with increasing periods of initial fog curing. However, if the fog curing period is terminated after 7, 14, or 28 days and controlled drying at 50 per cent relative humidity substituted, the concrete will develop many times greater resistance than companion concrete continuously fog cured to the same age.

2. Air-entrained concrete under moist freezing-and-thawing conditions absorbs water at a much lower rate than non-air-entrained concrete.

3. No appreciable effect was noted in the results obtained from the different methods of test performed on bars and cylinders due to changes in length of cycle or in the use of different freezing-and-thawing temperatures; however, large differences were observed between tests in which freezing of specimens was performed in different media, that is in water or in air.

4. Least variance in test results was obtained in the test No. 3 (slow water method) which consists of 24-hr periods of freezing at a temperature of 0 F and thawing at a temperature of 70 F.

5. In spite of the wide variation within tests, all methods of test investigated evaluated the different concretes in the same relative order.

6. Regardless of the test method, air-entrained concrete was much more durable than non-air-entrained concrete.

## REFERENCES

(1) W. H. Price and D. G. Kretsinger, "Aggregates Tested by Accelerated Freezing and Thawing of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 1108 (1951).

(2) Carl A. Menzel, "A Method for Determining the Moisture Condition of Hardened Concrete in Terms of Relative Humidity," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 1085 (1955).

(3) Joseph J. Shideler, "Investigation of the Moisture-Volume Stability of Concrete Masonry Units," *Bulletin No. D3*, Research and Development Lab., Portland Cement Assn., Chicago, Ill.

(4) L. E. Copeland and R. H. Bragg, "Self-Desiccation in Portland Cement Pastes," ASTM BULLETIN, No. 204, Feb., 1955, p. 34 (TP36).

(5) Effect of Various Specimen Storage Conditions During Freezing-and-Thawing Equipment Shut-Downs on the Durability of Concrete," *Report No. C-753*, Bureau of Reclamation, Concrete Lab., June, 1954.

(6) T. C. Powers, "Basic Considerations Pertaining to Freezing-and-Thawing Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 1132 (1955).

# FACTORS AFFECTING MEASUREMENT OF COMPRESSIVE STRENGTH OF INSULATING CONCRETES*

By Morton Sherman[1]

## Synopsis

For lack of experimental evidence to the contrary, testing laboratories and design engineers have found it necessary in the past to apply to lightweight insulating concretes the techniques, limits, and standard relationships that are proper for dense aggregate concretes. Some of these assumptions now appear to be erroneous when applied to insulating concretes.

Data presented in this study indicates that:

1. The effect of loading rate on the measured strength does not appear to be as critical as a variation in the dry density of the concrete under test.
2. Abnormal specimen height at casting can facilitate the formation of density gradients in the concrete specimen to be tested.
3. Without abnormal specimen heights, the height-width ratio of a test specimen can vary from 0.5 to 2.0 without seriously affecting the measured strength.
4. Improper curing can decrease the measured compressive strength.

A method of testing is suggested which utilizes 2-in. oven dry cubes.

Insulating concrete is defined in ASTM Specification C 332 – 56 T[2] as "concrete .... in which the prime consideration is the thermal insulating property of the resultant concrete."

This paper presents information concerning some of the variables affecting the measurement of compressive strength in insulating concretes, as uncovered in the course of work at the Zonolite Co.'s research laboratory at Evanston, Ill.

While the actual strength of insulating concretes is usually considered to be of secondary importance (**1,2**),[3] this physical characteristic is widely recognized as an excellent index of general concrete quality (**2**). It is used to determine the effect of variations in materials or conditions of manufacture, storage, or testing on many other properties of concrete. Compressive strength is also used to check the adequacy of mix design, to measure and control the quality of concrete, and to determine when part of a structure may be put in service.

Some of the factors which are known to affect the measured compressive strength are: cement content (quantity and composition), concrete density, curing conditions (temperature and humidity), curing time, specimen size and loading rate (**3–7**). Standards are necessary to control the effect of these and other variables, so as to evaluate properly the results of strength test data.

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*Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Chemical Engineer, Zonolite Co. Research Laboratory, Evanston, Ill.

[2] Tentative Specification for Lightweight Aggregates for Insulating Concrete (C 332 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 3, p. 167.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1109.

At the present time, there are two ASTM specifications which cover lightweight aggregates for insulating concrete[2] and for interior plaster.[4] Neither of these specifications makes reference to a standard test method for evaluating the compressive strength of such concretes or plasters.

TABLE I.—SPECIMEN SHAPE STUDY: MIX INFORMATION AND SUMMARY OF RESULTS.

MIX INFORMATION

| | 1:8 | 1:4 |
|---|---|---|
| Water, gal | 24 | 18 |
| Portland cement, lb | 94 | 141 |
| Stabilizing agent, weight oz | 12 | 6 |
| Vermiculite aggregate, lb | 64 | 48 |
| Mix time, min | 5 | 5 |
| Wet density, lb per cu ft | 50 | 57.5 |
| Slump, in. | 10 | 8.5 |
| Calculated expected dry density, lb per cu ft | 25.8 | 39.2 |

SUMMARY OF RESULTS

| | Compressive Strength, psi | Oven Dry Density, lb per cu ft | Compressive Strength, psi | Oven Dry Density, lb per cu ft |
|---|---|---|---|---|
| 2-in. cubes: | | | | |
| 17 psi per sec | 131 | 25.7 | 442 | 39.1 |
| 67 psi per sec | 151 | 26.1 | 465 | 39.7 |
| 4-in. cubes: | | | | |
| 4 psi per sec | 180 | 25.7 | 485 | 38.1 |
| 17 psi per sec | 167 | 25.7 | 489 | 38.2 |
| 29 psi per sec | ... | ... | 513 | 38.3 |
| 6 in. by 12-in. cylinders: | | | | |
| 2 psi per sec | 115 | 24.4 | ... | ... |
| 9 psi per sec | 108 | 24.4 | 446 | 36.5 |
| 16 psi per sec | ... | ... | 410 | 36.5 |

For this reason, design engineers, architects, and testing laboratories have had to rely on two principal sources for such testing procedures. These are: (1) raw material producers, and (2) existing specifications, originally designed for structural concretes, mortars and plasters.

Conflicts in technique between the specifications developed from experience with insulating concretes and the specifications which reflect dense concrete technology have been indicated in many published articles (**1,2,8,9**).

The selection of particular ASTM standards for application to insulating concretes has also been controversial, since this material could satisfy, under definitive conditions, the requirements of a concrete, a mortar, or a plaster. Those attempting to utilize specifications in this manner have been confronted with such problems as:

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[4] Tentative Specification for Inorganic Aggregates for Use in Interior Plaster (C 35 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 234.

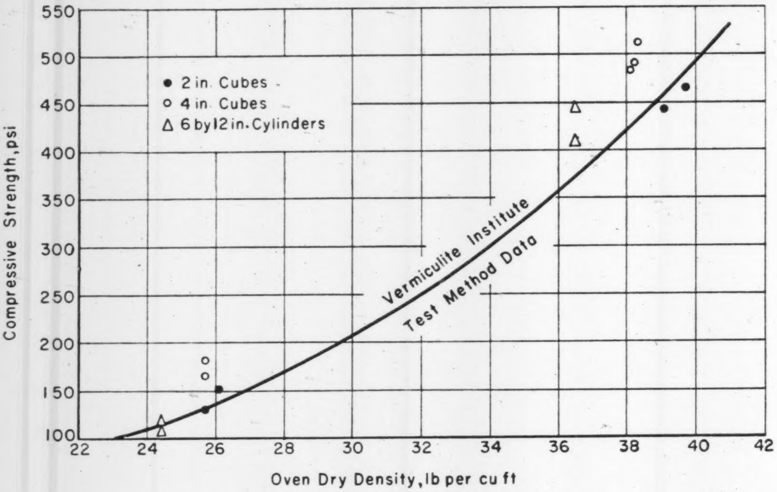

FIG. 1.—Comparison of Specimen Shape Study Results and Curve from Vermiculite Institute Test Method Data.

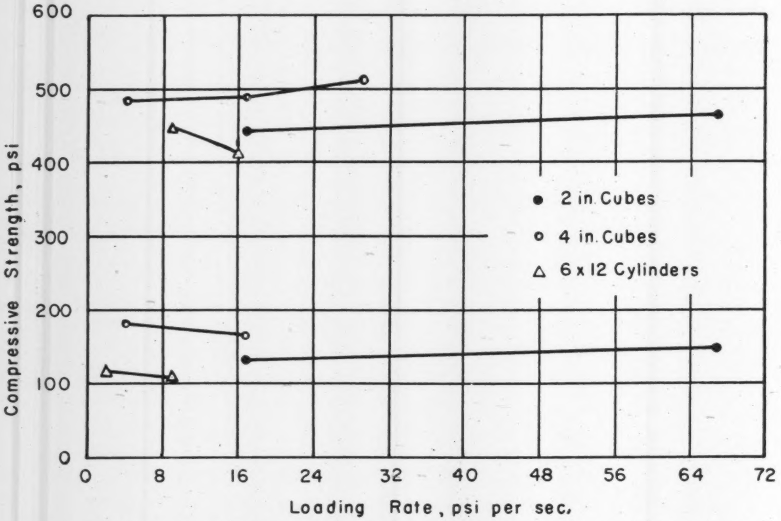

FIG. 2.—Specimen Shape Study: Measured Compressive Strength *versus* Uniform Loading Rate.

Shall 2-in. cubes be used in accordance with ASTM Method C 109 – 56,[5] or shall 3 by 6-in. or 6 by 12-in. cylinders be used in accordance with ASTM Method C 192 – 55?[6]

Shall specimens be cured by immersion in clear water as in ASTM Method C 109 – 56, by immersion in lime water as in ASTM Method C 192 – 55, or in a fog chamber?

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[5] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 56), 1956 Supplement to Book of ASTM Standards, Part 3, p. 129.

[6] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 55), 1955 Book of ASTM Standards, Part 3, p. 1318.

Should a loading rate of 20 to 50 psi per sec be used as in ASTM Method C 39 – 56 T,[7] or a loading time of 20 to 80 sec as in ASTM Method C 109 – 56?

Should a calculated corrected strength value that is unrealistic be accepted because the required correction factors are used as specified in ASTM Methods C 42 – 49?[8]

Should testing loads be applied to the top and bottom faces of the specimen, in accordance with ASTM Method C 39 – 56 T, or to the sides in accordance with ASTM Methods C 109 – 56, C 349 – 54 T,[9] C 257 – 52,[10] and C 26 – 56,[11] or merely reported as one or the other, in accordance with ASTM Methods C 42 – 49?

To aid in the resolution of some of these conflicts, a series of studies was undertaken by the Zonolite Co.'s research laboratory. Some of the variables investigated were specimen shape, uniform loading rates, specimen dimensions, and curing conditions.

## SPECIMEN DIMENSIONS AND LOADING RATE

One of the earliest studies undertaken was the investigation of specimen shape, size, and loading rate. Using 2-in. cubes, 4-in. cubes and 6 by 12-in. cylinders (a total of 120 specimens), this study utilized stabilized (air entrained) vermiculite insulating concrete in a density range of 25 to 40 lb per cu ft, at uniform loading rates of 2 to 67 psi per sec. Specimens were cured, oven dried to constant weight, and tested for compressive strength. General mix information and a summary of the results are shown in Table I.

From the data, it can be seen that:

1. The magnitude of compressive strength obtained using 6 by 12-in. cylinders was consistently lower than with either 2-in. or 4 in.-cubes. Significantly, cylinder dry density values were correspondingly lower than either 2-in. or 4-in. cubes (Fig. 1).

2. The effect of loading rate did not appear to be as great as the effect of dry density variation on the measured compressive strength, as high and low rates are on both sides of the average strength (Fig. 2).

In an attempt to evaluate the suitability of 6 by 12-in. specimens for compression testing, two such cylinders of 1:4 stabilized vermiculite insulating concrete were each sectioned into five horizontal levels, and four 2-in. cubes were taken from each level (Fig. 3). All 40 cubes were oven dried and weighed for dry density determinations, and then tested for compressive strength. The results of this study are given in Fig. 4.

There is an obvious correlation between compressive strength and density variations, indicating the presence of horizontal density and strength gradients within the molded cylinder. This would indicate the possibility that the measured compressive strength of 6 by 12-in. cylinders might reflect preferentially the strength of weaker, low density ends.

### *Specimen Height-Width Ratio:*

To evaluate the general effects of shape factor on the compressive strength

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[7] Tentative Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 3, p. 190.

[8] Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 – 49), 1955 Book of ASTM Standards, Part 3, p. 1360.

[9] Tentative Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (C 349 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 136.

[10] Method of Test for Compressive Strength of Magnesium Oxychloride Cements (C 257 – 52), 1955 Book of ASTM Standards, Part 3, p. 365.

[11] Methods of Testing Gypsum and Gypsum Products (C 26 – 56), 1956 Supplement to Book of ASTM Standards, Part 3, p. 26.

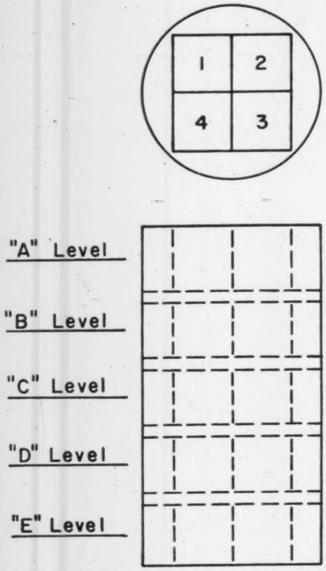


FIG. 3.—Sectioning of 6 by 12-in. Cylinders into 2 in. Cubes.

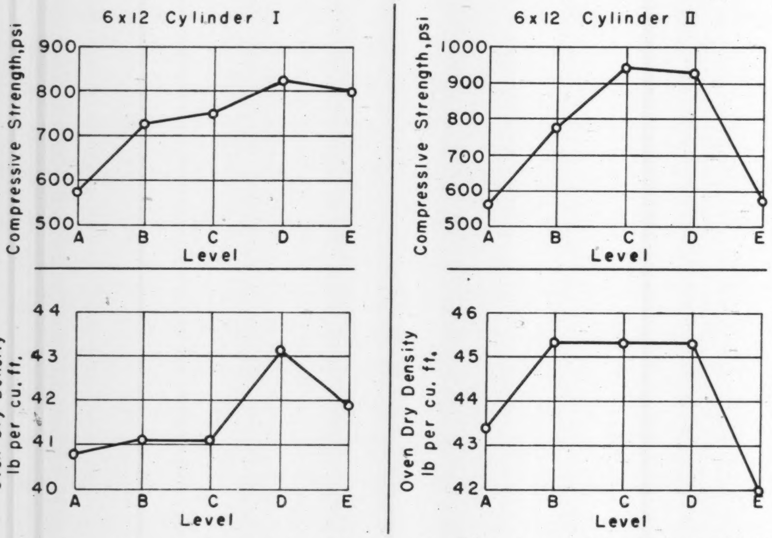


FIG. 4.—Comparison of Strength and Density Gradients in two 6 by 12-in. Cylinders.

of insulating concretes, further studies were undertaken wherein bearing plate contact area and specimen height were varied independently.

In the first part of the study, a series of specimens were cut from slabs of 1:4, 1:6, and 1:8 stabilized vermiculite insulating concrete, after curing 28 days in 73 F, 100 per cent relative humidity air. The prism specimens, over 200 in all, had a constant bearing plate contact area of 4 sq in. Specimen height varied from 1 to 4 in., resulting in a 0.5 to 2.0 range of height-width, or slenderness ratios. After oven drying, the specimens were tested at a loading rate of 17 psi per sec. Mix information and a summary of results are shown in Table II.

About 10 per cent of the compressive strength values fell outside of the ±10 per cent limits recommended in ASTM Method C 109 – 56, and were not used in determining the reported averages, which represent no fewer than six individual tests for each average.

A height-width, or slenderness ratio of 2.0, was considered to be "standard" in computing the experimental correction factors. Calculated factors ranged from

TABLE II.—HEIGHT-WIDTH RATIO STUDY: MIX INFORMATION AND SUMMARY OF RESULTS.

MIX INFORMATION

| | 1:4 Mix | 1:6 Mix | 1:8 Mix |
|---|---|---|---|
| Water, gal | 12.5 | 12.5 | 13 |
| Portland cement, lb | 94 | 63 | 47 |
| Stabilizing agent, weight, oz | 4 | 4 | 4 |
| Vermiculite aggregate, lb | 32 | 32 | 32 |
| Wet density, lb per cu ft | 51.3 | 46.3 | 42.5 |

SUMMARY OF RESULTS

| Mix | Specimen Height, in. | Height-Width Ratio | Average Oven Dry Density, lb per cu ft | Average 28-day Compressive Strength, psi | Calculated Height-Width Correction Factor | ASTM Structural Concrete Correction Factor |
|---|---|---|---|---|---|---|
| 1:4 | 1 | 0.5 | 38.5 | 380 | 1.07 | 0.50 |
| | 2 | 1.0 | 37.3 | 387 | 1.04 | 0.85 |
| | 3 | 1.5 | 37.0 | 387 | 1.05 | 0.96 |
| | 4 | 2.0 | 37.0 | 405 | 1.00 | 1.00 |
| 1:6 | 1 | 0.5 | 28.6 | 185 | 1.01 | 0.50 |
| | 2 | 1.0 | 28.8 | 188 | 0.99 | 0.85 |
| | 3 | 1.5 | 28.8 | 194 | 0.96 | 0.96 |
| | 4 | 2.0 | 28.8 | 186 | 1.00 | 1.00 |
| 1:8 | 1 | 0.5 | 23.6 | 108 | 0.99 | 0.50 |
| | 2 | 1.0 | 22.1 | 103 | 1.04 | 0.85 |
| | 3 | 1.5 | 22.5 | 102 | 1.05 | 0.96 |
| | 4 | 2.0 | 22.5 | 107 | 1.00 | 1.00 |

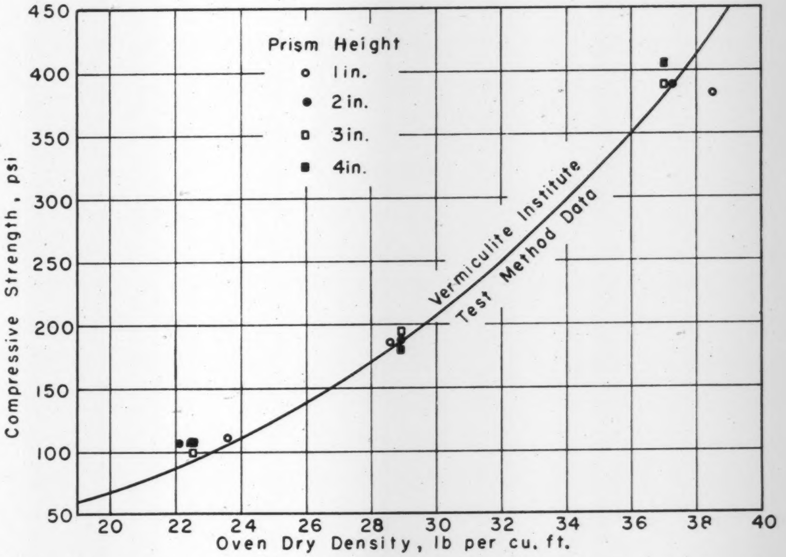


FIG 5.—Specimen Dimension Study; Variable Specimen Height, Constant Bearing Plate Contact Area, Constant Loading Rate.

TABLE III.—BEARING PLATE CONTACT AREA STUDY: SUMMARY OF RESULTS.

| Prism Dimensions, in. | Contact Area, sq in. | Height-Width Ratio | Measured Compressive Strength, psi | Calculated Height-width Correction Factor[a] |
|---|---|---|---|---|
| 1 by 1 by 3 | 1 | 3 | 239 | 1.01 |
| 2 by 2 by 3 | 4 | 1.5 | 244 | 0.99 |
| 3 by 3 by 3 | 9 | 1 | 259 | 0.93 |
| 4 by 4 by 3 | 16 | 0.75 | 268 | 0.90 |
| 5 by 5 by 3 | 25 | 0.6 | 275 | 0.88 |

[a] Extrapolated strength of standard 2.0 H/W ratio = 242 psi

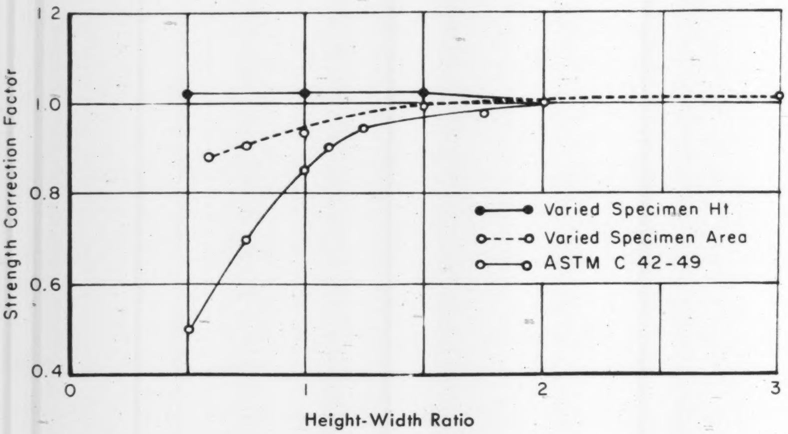


FIG. 6.—Calculated Height-Width Ratio Strength Correction Factors as Compared to ASTM C 42–49.

−4 per cent to +7 per cent of the standard, a negligible variation when compared to the −4 per cent to −50 per cent factors specified in ASTM C 42 – 49.

Figure 5, a plot of the strength data, indicates the conformity to the same "standard conditions" curve shown in Fig. 1. Note the close grouping of the data.

In the second part of the study, specimens were tested wherein the height was held constant and bearing plate contact area became the variable. Over 50 specimens, cut from a 3 inch thick slab of 1:6 stabilized vermiculite concrete having an oven dry density of 30 lb per cu ft after extended curing under ordinary room conditions of temperature and humidity, and tested at a 17 psi per sec loading rate, were used to obtain the data shown in Table III.

Table III indicates the possibility of increase in measured compressive strength with increasing bearing plate contact. It should be noted, however, that all the test data is within ±10 per cent of the average. This would indicate that the relationship, if real, is of a low order of magnitude.

Figure 6 suggests that specimen dimensions can be considered a minor variable, and not critical, within reasonable limits, in the testing of insulating concretes for compressive strength. Of all the variables investigated up to this point, only oven dry density appears to have a significant relation to the measured compressive strength. Where sample size could cause density variations, as in the case of abnormally tall specimens, significant changes could be experienced.

TABLE IV.—CURING HUMIDITY STUDY: CURING TIME *versus* COMPRESSIVE STRENGTH.

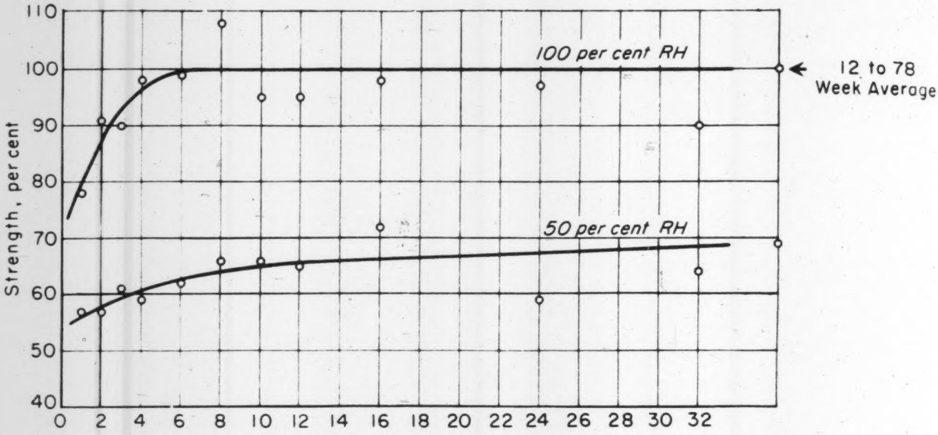
| Curing Time, weeks | Compressive Strength at Three Oven Dry Densities | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 34 lb per cu ft | | | | 26 lb per cu ft | | | | 21 lb per cu ft | | | |
| | 50% RH | | 100% RH | | 50% RH | | 100% RH | | 50% RH | | 100% RH | |
| | psi | per cent[a] | psi | per cent[a] | psi | per cent[a] | psi | per cent[a] | psi | per cent[a] | psi | per cent[a] |
| 1 | 198 | 57 | 270 | 78 | 97 | 56 | 113 | 65 | 57 | 51 | 64 | 58 |
| 2 | 196 | 57 | 313 | 91 | 96 | 55 | 136 | 78 | 65 | 59 | 72 | 65 |
| 3 | 211 | 61 | 311 | 90 | 102 | 59 | 154 | 89 | 69 | 62 | 93 | 84 |
| 4 | 203 | 59 | 339 | 98 | 107 | 62 | 141 | 81 | 67 | 60 | 82 | 74 |
| 6 | 214 | 62 | 343 | 99 | 100 | 58 | 158 | 91 | 68 | 61 | 94 | 85 |
| 8 | 228 | 66 | 373 | 108 | 111 | 64 | 174 | 100 | 69 | 62 | 98 | 88 |
| 10 | 226 | 66 | 326 | 95 | 103 | 59 | 158 | 91 | 62 | 56 | 118 | 106 |
| 12 | 224 | 65 | 327 | 95 | 103 | 59 | 186 | 107 | 64 | 58 | 105 | 95 |
| 16 | 248 | 72 | 338 | 98 | 110 | 63 | 192 | 110 | 70 | 63 | 119 | 107 |
| 24 | 204 | 59 | 333 | 97 | 91 | 52 | 163 | 94 | 62 | 56 | 107 | 96 |
| 32 | 221 | 64 | 310 | 90 | 103 | 59 | 158 | 91 | 63 | 57 | 117 | 105 |
| 40 | 268 | 78 | 394 | 114 | 125 | 72 | 186 | 107 | 72 | 65 | 118 | 106 |
| 48 | 266 | 77 | 372 | 108 | 114 | 66 | 167 | 96 | 71 | 64 | 111 | 100 |
| 78 | 232 | 67 | 343 | 99 | 108 | 62 | 168 | 97 | 62 | 56 | 110 | 99 |
| Average 12 to 78 weeks | 238 | 69 | 345 | 100 | 108 | 62 | 174 | 100 | 66 | 60 | 111 | 100 |

[a] Per cent of average strength for 100 per cent relative humidity curing during the 12 to 78 week period.

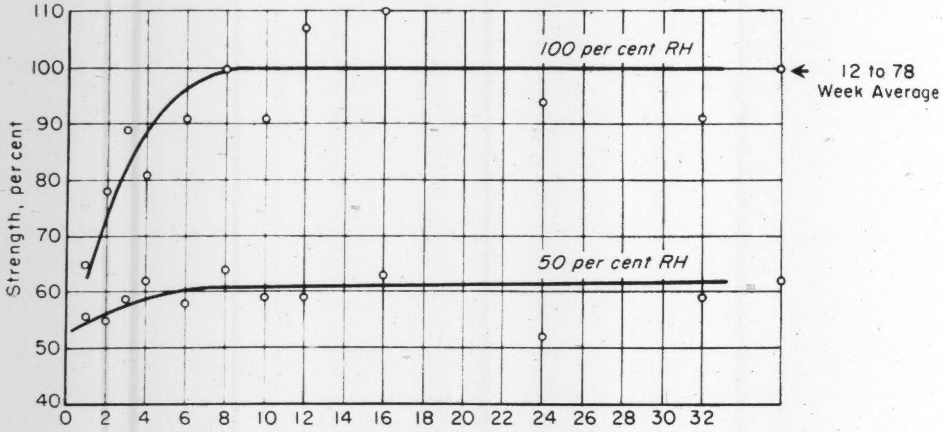
Anderegg (**8**) reported generally similar results in a study on moist cured perlite concrete, where strength values obtained using 2-in. cubes, 3 by 6-in. and 6 by 12-in. cylinders generally agreed within ±5 per cent. In the same presentation, data were given for 2-in. cubes, 2 by 2 by 4-in. and 2 by 2 by 6-in. prisms showing a maximum of 9 per cent variation between shapes, or less than 5 per cent from the average.

Gyengo (**10**), Tucker (**11**), and more recently Murdock and Kesler (**12**) are among the many who have reported differences in the measured compressive strength of dense concretes and mortars when tested as cubes, modified cubes, or cylinders. The latter authors have made a comprehensive study of this effect and indicate that correction factors should vary with concrete strength, as no one set of factors will be correct for all concretes, and the influence of aggregate type might require the use of individual correction factors.

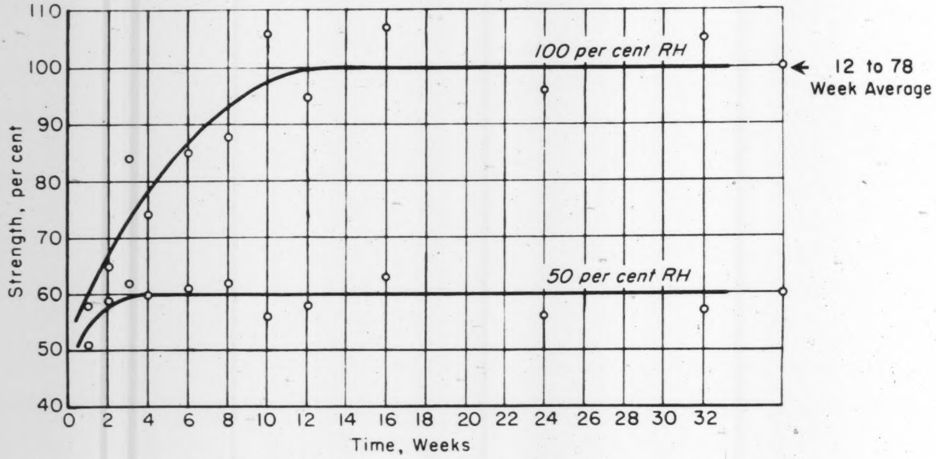
This thought is also borne out by Rusch (**9**), who believes the effect to be a systematic problem, where hard materials could produce end plate distortions, while relatively weaker lightweight concretes would not. Anderegg (**8**) stated that lightweight concretes might actually follow a "different law" under loading. The absence of the familiar double cone in failure and the apparent tendency for



(*a*) Compressive strength of 34 lb per cu ft oven dry density concrete.



(*b*) Compressive strength of 26 lb per cu ft oven dry density concrete.



(*c*) Compressive strength of 21 lb per cu ft oven dry density concrete.

FIG. 7.—Curing Study; Compressive Strength of Vermiculite Concrete, Expressed as Per Cent of 12 to 78 Week, 100 Per Cent Relative Humidity Average.

localized failure points are given as evidence supporting the theory.

## CURING

In order to obtain uniform and reproducible compressive strength measurements with concrete specimens, standard curing conditions are necessary. It is common knowledge that both temperature and humidity affect the strength attainment of portland-cement products, especially in the early stages.

According to Powers (**5**), to obtain the maximum possible rate of hydration, at constant temperature, concrete must be kept saturated with water. Soon after test specimens are stored at room humidity, the cement apparently ceases to hydrate. This is shown by the lack of strength-gain, except for that due to drying. Price (**6**) amplifies this general statement with supporting data, showing that continued moist curing results in greater compressive strength, and that richer mixes gain strength faster than lean mixes when cured under water.

Increasing the curing temperature increases the rate of cement hydration and therefore affects the rate of strength development (**2**, **13**).

To evaluate the effect of curing atmosphere humidity variations on insulating concrete compressive strength, three stabilized vermiculite insulating concretes, ranging from 21 to 34 lb per cu ft oven dry density, were cured at 73 F over a 78 week period, in 50 per cent and 100 per cent relative humidity atmospheres.

At least three, and in most cases six, individual specimens were tested to obtain the results shown in Table IV and Fig. 7 (*a*) (*b*) and (*c*). These results are expressed as measured compressive strength in psi, and the relative strength as compared to the average strength obtained with moist curing over the 12 to 78-week interval (noted as the percentage of this average strength).

The following observations can be made from the results of this study:

1. The rate of attainment of compressive strength increases as the oven-dry density, and therefore cement content, increases. This is found to be the case at both 50 per cent and 100 per cent relative humidity.
2. At the end of 28 days of moist curing, only the densest (high cement content) insulating concrete has reached its apparent maximum. The lowest density (low cement content) insulating concrete appears to require about 3 months of moist curing to reach a maximum value.
3. After 4 months of curing have elapsed, strength becomes relatively constant for all concretes cured at 100 per cent relative humidity. Only with low humidity curing and high cement content does strength still increase with time.

In every test series there appears to be a "cycling" pattern to the strength data. This might be attributed to seasonal changes in humidity in the room where specimens were cooled to room temperature after oven drying. The test series was started in February, and during the first 16 weeks, low room humidities had no serious effect on measured strength. Measurements taken on the 24th and 32nd weeks (July and September) were generally lower than those taken before or after this high-humidity period. Significantly, high measurements were generally obtained during the 40th and 48th week (November and January) low humidity interval. Measurements taken on the 78th week (July) were again low. Averaged over the 12 to 78 week or 24 to 78 week intervals, there seems to be no significant change in strength with time after the 12th week. Two or more complete seasonal cycles would be required to confirm these preliminary conclusions.

4. Immersion curing was also studied in this series (data not shown) and gave strength results virtually the same as

did 100 per cent relative humidity curing. Higher density (richer) concrete had slightly higher strength with immersion curing as compared to humid air curing, while lower density (leaner) concrete tended to show slightly poorer strength with immersion curing.

The data obtained in this study appears to be in general agreement with the technology of dense concretes, as stated in the American Concrete Inst., "Manual of Concrete Inspection" (**13**), as discussed by Price (**6**), Kesler and Seiss (**2**), and many others.

## Moisture Content

Gonnerman (**3**), Gilkey (**14**), Lea and Desch (**15**), and Bessey and Dilnot (**16**) indicate that the effect of moisture content on compressive strength is characteristic of both dense and lightweight concretes.

Gonnerman (**3**) indicates the desirability of basing strength determinations and comparisons on a standard moisture content and suggests two possible test conditions—thoroughly saturated and thoroughly dried. The saturated or soaked condition, because of the difficulty in drying dense concrete specimens to a constant weight in a short period of time, has become the accepted testing procedure for conventional concrete. If there were not the difficulty in drying large, dense specimens to constant weight, it would probably be more desirable to test these concretes in the dry condition, to reflect the strength increase due to moisture loss after curing.[12]

Bessey and Dilnot (**16**) indicate that the decrease in strength of cellular concretes due to the presence of the first 2 or 3 per cent of "free moisture," or evaporable water, is considerable and constitutes a high proportion of the total decrease in strength from the dry to the wet condition. This information was obtained in a study on material which had been either steam cured at 65 C and dried to constant weight at 140 C, or autoclaved at 183 C and cooled.

It is recognized that higher hydration temperatures such as these, especially early in the curing history of a concrete, modify the physical characteristics of the portland-cement binder. At ordinary temperatures the hydration products form principally as a gel, whereas at elevated temperatures they become microscopically crystalline. The effect is progressive—the higher the temperature, the smaller the apparent amount of gel per unit of hydrated cement (**13**). Such modification also tends to lower the ultimate strength of an ordinary portland-cement paste, and decrease its drying shrinkage. With such radical changes in the physical properties at high curing temperatures, we could also expect the surface area available for water vapor adsorption, the weight per cent of water vapor adsorbed at a given humidity condition, and therefore the effect of such adsorbed water on the measured compressive strength, to be significantly changed also.

Studies in progress at the Zonolite Co. laboratory indicate that this sharp change in strength with low moisture content, as reported by Bessey and Dilnot, is not observed in the case of moist-cured vermiculite insulating concretes. However, the study is not complete, and test data cannot be reported at this time.

Valore (**1**) refers to the current practice of testing some insulating concretes after they have been oven dried and suggests testing at 28 days after curing in air at ordinary temperatures.

In the evaluation of concrete quality, as indicated by the magnitude of compressive strength attained by a particular concrete mix, it is desirable that as much moist curing as possible (within reasonable testing time limitations) be given specimens under test. ASTM Methods C 109 - 56,[5] C 349 - 54 T,[9] C 31 - 55[13] and C 192 - 55[6] all recommend continuous curing in the range of 65 to 75 F in a moist condition until the time of test.[8]

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[12] Am. Concrete Inst., Committee 704, Specification for Cast Stone, *Proceedings*, Am. Concrete Inst., Vol. 41, June, 1945, pp. 701–703.

A short moist curing interval could conceivably result in compressive strengths half the order of magnitude of strengths obtained with longer curing (6). A curing time of 28 days has been accepted as representative of many construction requirements; for quality evaluation, an extended curing period would not be practical.

To oven dry the generally smaller, lighter weight and more permeable specimens of insulating concrete is not inconvenient, and allows a maximum of moist curing within a practical testing time.

The Vermiculite Institute has adopted as an industry standard a procedure for the sampling and compression testing of vermiculite lightweight concrete for roof fills, roof decks, and floors (17). This is an interim standard, pending the development of a standard testing method by ASTM Committee C-9, on Concrete and Concrete Aggregates. In brief, this procedure calls for:

1. Three or more 2-in. cubes made in standard cube molds or cut from 2-in. thick cast slabs after curing.
2. Moist curing for 28 days in a 73 F, 100 per cent relative humidity atmosphere.
3. Drying to constant weight at 220 to 230 F.
4. Load applied to the top and bottom of each cube; clay-sulfur capping techniques when required.
5. Loading time to failure, 20 to 80 sec per specimen.
6. Report to indicate individual tests and average oven dry density and compressive strength.

Figure 8 indicates the results obtained

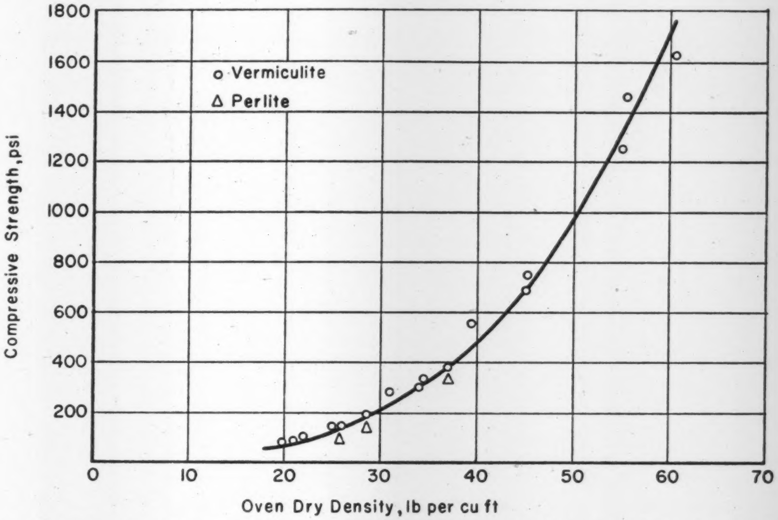

FIG. 8.—Compressive Strength *versus* Oven Dry Density, Using Vermiculite Institute Testing Specifications.

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[13] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 - 55), 1955 Book of ASTM Standards, Part 3, p. 1314.

when 16 vermiculite and three perlite concretes, covering an oven dry density range of 20 to 60 lb per cu ft, were cured and tested in this manner.

## SUMMARY

In summary, the data appears to indicate that test specimen dimensional relationships, and rate of application of load are not critical, when kept within reasonable limits. Extent of moist curing curing, temperature, moisture content at the time of test, and oven dry density (indicative of cement content), can all seriously affect the magnitude of measured compressive strength.

Continued research into the latter variables, to determine definitely and accurately the magnitude of their interrelationships, appears imperative. Other matters, such as the evaluation of various capping techniques (**1**,**2**), "aelotropy" (**18**), aggregate hardness (**19**), the relationship between inherent binder strength (as measured according to ASTM Method C 109 - 56) and the resultant insulating concrete strength-density ratio, etc., offer real promise in further extending and increasing our basic knowledge of the strength characteristics of insulating concretes.

## REFERENCES

(1) R. C. Valore, Jr., "Insulating Concretes," *Proceedings*, Am. Concrete Inst., Vol. 53, Nov., 1956, pp. 509–532.

(2) R. E. Davis and J. W. Kelly, "Lightweight Concrete and Aggregates," Symposium on Significance of Tests of Concrete, Am. Soc. Testing Mats., pp. 238–249. (1955). (Issued as separate publication *ASTM STP No. 169*).
S. B. Helms, "Air Content and Unit Weight," Am Soc. Testing Mats. *ASTM STP No. 169*, pp. 208–220 (1956).
C. E. Kesler and C. P. Seiss, "Static and Fatigue Strength," Am. Soc. Testing Mats. *ASTM STP No. 169*, pp. 81–93 (1956).

(3) H. F. Gonnerman, "Study of Methods of Curing Concrete," *Proceedings*, Am. Concrete Inst., Vol. 26, Feb., 1930, p. 359.

(4) H. F. Gonnerman, and Wm. Lerch, "Changes in Characteristics of Portland Cement as Exhibited by Laboratory Tests over the Period 1904 to 1950." Am. Soc. Testing Mats. (1951). (Issued as separate publication *ASTM STP No. 127*).

(5) T. C. Powers, "A Discussion of Cement Hydration in Relation to the Curing of Concrete," *Proceedings*, Highway Research Board, 27th Annual Meeting, Dec., 1947, pp. 178–188.

(6) W. H. Price, "Factors Affecting Concrete Strength," *Proceedings*, Am. Concrete Inst., Vol. 47, Feb., 1951, pp. 417–432.

(7) R. C. Valore, Jr., "Cellular Concretes," *Procedings*, Am. Concrete Inst., Vol. 50, May–June, 1954, pp. 773–796, 817–836.

(8) F. O. Anderegg, "Shape Factor and Compressive Strength," *Proceedings*, Am. Concrete Inst., Vol. 49, Dec., 1952, pp. 333–334 (LR-49-10).

(9) H. Rusch, "Specimen Size and Apparent Compressive Strength," *Proceedings*. Am. Concrete Inst., Vol. 50, May, 1954, pp. 803–804 (LR-50-18).

(10) T. Gyengo, "Effect of Type of Test Specimen and Gradation of Aggregate on Compressive Strength of Concrete," *Proceedings*, Am. Concrete Inst., Vol. 33, Jan–Feb., 1938, pp. 269–282.

(11) J. Tucker, Jr., "Effect of Length on the Strength of Compression Test Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 976 (1945).

(12) J. W. Murdock and C. E. Kesler, "Effect of Length to Diameter Ratio on the Apparent Compressive Strength of Concrete," ASTM BULLETIN, No. 221, Apr., 1957, pp. 68–73 (TP76).

(13) "Manual of Concrete Inspection," Am. Concrete Inst., Third Edition, pp. 20–24 (1955).

(14) H. J. Gilkey, "Discussion—Study of Methods of Curing Concrete," *Proceedings*, Am. Concrete Inst., Vol. 26, June, 1930, pp. 898–901.

(15) F. M. Lea and C. H. Desch, "The Chemistry of Cement and Concrete," E. Arnold & Co., London, p. 233 (1940).

(16) G. E. Bessey, and S. Dilnot, "Relation Between Strength and Free Water Content of Aerated Concretes," *Magazine of Concrete Research* (London) Dec., 1949, pp. 119–122.

(17) "Procedure for Sampling and Compression Testing of Vermiculite Lightweight Concrete for Roof Fills, Roof Decks and Floors," *Concrete Bulletin*, Vermiculite Inst., No. 87-A, March, 1957.

(18) L. B. Mercer, "Concrete—An Aelotropic Material," *Commonwealth Engineer* (Melbourne), Jan. 2, 1956, pp. 183–188.

(19) J. J. Brouk, "Perlite Insulating Concrete," *Proceedings*, Am. Concrete Inst., Vol. 50, June, 1954, pp. 857–867.

(20) "Heating, Ventilating, Air Conditioning Guide," Am. Soc. Heating, Air Conditioning Engineers, Vol. 33, p. 171 (1955).

(21) "Permalite Specification Manual," Great Lakes Carbon Corp., Perlite Division, Los Angeles, Calif. (1954).

(22) "The Mearlcrete System for Moist Cured Foam Concrete," Mearl Mfg. Corp., Roselle Park, N. J. (1956).

(23) S. Thaulaw, "Tensile Splitting Test and High Strength Concrete Test Cylinders," *Proceedings*, Am. Concrete Inst., Vol. 53, Jan. 1957, pp. 699–706.

(24) "Lightweight Aggregates," Housing and Home Finance Agency, U.S. Govt. Printing Office, Washington, D. C., Aug. 1949.

# DISCUSSION

Mr. S. B. Helms[1] (*presented in written form*).—Two subcommittees of ASTM Committee C-9 on Concrete and Concrete Aggregates are now actively working on the testing of lightweight aggregate concrete for strength and the author's results indicate that special procedures should be established for making and testing of insulating concrete specimens. It was explained to the author in February 1956 that a cooperative test series was to be undertaken to reappraise the accuracy of the length-diameter correction factors given in ASTM Methods C 42.[2] Extensive test work on vermiculite aggregate concrete, which was completed years ago under the direction of J. C. Pearson, had prompted Mr. Pearson to discuss J. W. Johnson's article which appeared in January 1943 ASTM Bulletin.[3] He commented on the unusual character of low-density concretes which are weak in strength. These ideas suggested that representatives of the vermiculite industry should be invited to report on their experiences in making strength tests of insulating concrete.

The data in the author's Table II covering the range of volume proportions 1:4 to 1:8 and strengths in the range of 100 to 400 psi support his statement that the height-width ratio can vary from 0.5 to 2.0 without seriously affecting the measured strength. Upon receipt of a copy of the paper it was decided to undertake a brief series of tests with two aggregate samples and obtain confirmatory data using type III cement. Use of high early strength cement was essential in order to complete the tests in time for the results to be available when the paper was presented. Our test data were developed specifically for the two Committee C-9 subcommittees but are of interest in connection with the present paper. While the Zonolite results in Table II are based on prisms sawed from slabs, our approach was to saw slices of varying height from uniformly molded prism specimens which had cross-sections of 2 by 2 in. and 3 by 3 in. respectively.

Limitations of time and materials precluded the making of preliminary batches with both aggregate samples which were recently received and therefore the expanded perlite sample was selected for use in trial concrete batches.

*Preliminary Tests:*

The plan of the experiment was to make four test mixes and mold the same assorted sizes of specimens from each mix. These sizes were: 2-in. cubes, 2 by 4-in. and 3 by 6-in. cylinders, 2 by 2 by 10-in. and 3 by 3 by 15-in. prisms, all in metal molds, plus a few extra 3 by 6-in. cylinders made in paper molds for a side test of the effect of oven drying.

Test concretes containing each aggre-

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[1] Research Engineer, Lehigh Portland Cement Co., Allentown, Pa.

[2] Methods of Securing, Preparing and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 – 49), 1955 Book of ASTM Standards, Part 3, p. 1360.

[3] James W. Johnson, "Effect of Height of Test Specimens on Compressive Strength of Concrete," ASTM Bulletin, No. 120, Jan., 1943, p. 19.

TABLE V.—UNIFORMITY OF INSULATING CONCRETE CAST IN 6 BY 12-IN. MOLDS.

Type III cement-expanded perlite mixes containing air entraining agent.

Sawed 4 in. Cubes Representing Three Levels.

| | Mix 1T | Mix 2T |
|---|---|---|
| Dry Volume Proportions | 1:4 | 1:6 |
| Slump, in. | 8¼ | 8¼ |
| Moist Cured, days | 3 | 2 |
| AIR DRY DENSITY AT AGE 7 DAYS, LB PER CU FT | | |
| Top | 36.0 | 25.5 |
| Middle | 35.7 | 25.0 |
| Bottom | 35.8 | 25.1 |
| Average Air Dry Density | 35.8 | 25.2 |
| Calculated Oven Dry Density | 30.3 | 22.5 |
| AIR DRY COMPRESSIVE STRENGTH OF 4-IN. CUBES AT AGE 7 DAYS, PSI | | |
| Top | 415 | 165 |
| Middle | 404 | 140 |
| Bottom | 409 | 140 |
| Average | 409 | 148 |

gate were proportioned 1:4 and 1:6 by volume and the series consisted of mechanically mixed batches made in two rounds. The first round of specimens was tested in the air-dry state after a 5 day moist cure and the second round of specimens were moist cured 7 days and tested in the undried state.

Preliminary perlite aggregate trial batches containing type III cement and air entraining admixture were made according to the manufacturer's recommendations to establish a suitable sequence for obtaining uniform mechanically mixed batches. These mixes are shown as 1T and 2T in the accompanying Table V, and were similar in composition to test batches Nos. 1 and 2 which will be described later.

One 6 by 12-in. cylinder was made from each trial batch and after a short cure these cylinders were sawed into three equal slices which were finally cut to make 4-in. cube specimens. The volume of cubes was determined by displacement before drying started. Table V shows that these cylinders, made from 1:4 and 1:6 air-entrained perlite mixes of soft consistency, were reasonably uniform in density and compressive strength. It is possible that richer perlite aggregate mixes, with greater density, would show less "top to bottom" uniformity. However, it seemed advisable to show the results for these two 6 by 12-in. cylinders as incidental information.

*Test Mixes:*

The sequence of addition of constituent materials to the batches should be mentioned. About 85 per cent of the total estimated water was used to make a slurry of the cement, with air entraining agent included, and after any lumps were smoothed out the dry aggregate was introduced. Following a final addition of water to reach the desired consistency the batch was mixed for a period of 2 min.

The various sizes of specimens were molded at one time by three technicians who used appropriate methods of consolidation. Table VI fully describes the batches made for both test rounds, but for brevity the second slide includes only the mix data for the first round. Use of the same proportion of air entraining agent in these batches resulted in concretes of similar cement content with each aggregate. While the $1\frac{1}{4}$ oz dry admix per sack of aggregate resulted in nearly optimum placeability for perlite mixes, the amount of entrained air was judged insufficient in the case of both vermiculite mixes. In the second round the amount of admixture in both vermiculite batches was increased to $2\frac{1}{2}$ oz per sack of aggregate and we found that

TABLE VI.—COMPOSITION AND PROPERTIES OF TEST MIXES, LIGHTWEIGHT AGGREGATE INSULATING CONCRETES.

| Round I Test | Batch No. 1 | Batch No. 2 | Batch No. 3 | Batch No. 4 |
|---|---|---|---|---|
| Aggregate Type<br>Volume Proportions<br>Air Entraining Agent, oz per 4 cu ft | Perlite<br>1:4<br>1.25 | Perlite<br>1:6<br>1.25 | Vermiculite<br>1:4<br>1.25 | Vermiculite<br>1:6<br>1.25 |
| Quantities, lb per cu ft | | | | |
| Type III cement | 19.6 | 11.9 | 18.7 | 11.1 |
| Air Entraining admix (dissolved) | 0.22 | 0.20 | 0.21 | 0.19 |
| Water | 15.9 | 12.9 | 26.3 | 21.6 |
| Aggregate | 8.0 | 7.2 | 8.4 | 7.4 |
| Total (fresh density) | 43.7 | 32.2 | 53.6 | 40.3 |
| Measured slump, 12 in. cone | 8.2 in. | 8.4 in. | 6.6 in. | 7.8 in. |
| 6 in. cone | 2.6 in. | 3.4 in. | 1.8 in. | 2.4 in. |
| Cement content, sacks per cu yd | 5.6 | 3.4 | 5.4 | 3.2 |
| Aggregate content, cu ft per cu yd | 22.5 | 20.5 | 21.5 | 19.1 |
| Gross water content, gal per sack | 9.1 | 12.3 | 15.8 | 19.5 |

| Round II Test | Batch No. 5 | Batch No. 6 | Batch No. 7 | Batch No. 8 |
|---|---|---|---|---|
| Air Entraining Agent (oz per 4 cu ft) | 1.25 | 1.25 | 2.50 | 2.50 |
| Quantities, lb per cu ft | | | | |
| Type III cement | 19.5 | 12.2 | 15.5 | 11.2 |
| Air Entraining admix (dissolved) | 0.22 | 0.20 | 0.35 | 0.38 |
| Water | 15.8 | 13.2 | 21.1 | 23.6 |
| Aggregate | 8.0 | 7.4 | 6.9 | 7.5 |
| Total (fresh density) | 43.5 | 33.0 | 43.8 | 42.7 |
| Measured slump, 12 in. cone | 8.0 in. | 8.2 in. | 8.3 in. | 5.8 in. |
| 6 in. cone | 2.6 in. | 3.3 in. | 3.5 in. | 1.1 in. |
| Cement content, sacks per cu yd | 5.6 | 3.5 | 4.4 | 3.2 |
| Aggregate content, cu ft per cu yd | 22.5 | 21.0 | 17.8 | 19.3 |
| Gross water content, gal per sack | 9.4 | 12.3 | 15.4 | 23.7 |

this dosage entrained substantially more air in the 1:4 vermiculite mix but the fresh density of the 1:6 vermiculite mix was not reduced. No explanation of this behavior is given since we did not repeat batch No. 8. Usually the manufacturer of vermiculite incorporates the "stabilizer" in the aggregate in the recommended proportion but we thought it desirable to add the agent in solution to insure uniform dispersion. The respective perlite aggregate mixes of round II were very similar to batches No. 1 and No. 2 showing that the same level of air content was attained.

TABLE VII.—INDIVIDUAL UNCORRECTED COMPRESSIVE STRENGTH VALUES.

Specimens saved to study effect of height-width ratio.
Slices sawed from bars of two sizes. Two test conditions for expanded perlite aggregate concrete.

| Mix Proportions | Cross-Section, in. | Compressive Strength for Four Height-Width Ratios, psi | | | | Average, All Height-Width Ratios |
|---|---|---|---|---|---|---|
| | | 2.00 | 1.00 | 0.75 | 0.50 | |
| PERLITE CONCRETE TESTED AIR DRY AT AGE 9 DAYS AFTER 5 DAY CURE | | | | | | |
| 1:4 | 2 by 2 | 533 | 629 | 599<br>679 | 583 | 604 |
| 1:4 | 3 by 3 | 531 | 489 | 597 | 584<br>579 | 556 |
| 1:6 | 2 by 2 | 177 | 246 | 200 | 216<br>232 | 214 |
| 1:6 | 3 by 3 | 174 | 154 | 194 | 194<br>194 | 182 |
| PERLITE CONCRETE TESTED MOIST AFTER 7 DAY CURE | | | | | | |
| 1:4 | 2 by 2 | 526 | 554 | 551<br>581 | 556 | 554 |
| 1:4 | 3 by 3 | 419 | 434 | 388<br>385 | 404 | 406 |
| 1:6 | 2 by 2 | 158 | 168 | 157<br>143 | 175 | 160 |
| 1:6 | 3 by 3 | 141 | 144 | 139 | 148<br>135 | 141 |
| Average 1:4 mixes | | 502 | 526 | 540 | 541 | 530 |
| Average 1:6 mixes | | 162 | 178 | 167 | 185 | 174 |

*Molding Test Specimens:*

All mixes were of soft consistency and easy to place in the molds. Slump and fresh unit weight (yield) tests were completed prior to making any specimens; thus the slumps at time of placing were less than the measured values reported. All mixes had a cohesive nature attributed to the entrained air and high early cement.

Rodding with metal tools was not a satisfactory procedure because hidden voids would be likely to form and heavy tools are unwieldy. Lightweight tampers, such as specified in making C 109 mortar cubes, resulted in satisfactory consolidation but tended to increase density by compaction. Small rectangular scoops and bladed tools, such as spatulas or rectangular flat metal strips, proved to be satisfactory in molding prisms since they facilitated consolidation by puddling without tending to introduce voids which would render the specimens non-uniform. Examination of sawed slices from prisms for height-width ratio tests did not reveal any trapped voids worth considering.

*Effect of Height-Width Ratio in Insulating Concrete:*

Compressive strength data of individual specimens are listed in Tables VII and VIII. The average values have been added in a column at the right edge of Tables IX and X, to indicate the strength levels at which the tests were made. Presentation of relative strength data is about the simplest way to show the effect of height-width ratio in the case of these low density concretes. The average relative strength for various levels is considered to be the most reliable index in a planned experiment requiring tests of single specimens.

TABLE VIII.— INDIVIDUAL UNCORRECTED COMPRESSIVE STRENGTH VALUES.

Specimens sawed to study effect of height-width ratio.
Slices sawed from bars of two sizes. Two test conditions for vermiculite aggregate concrete.

| Mix Proportions | Cross-Section, in. | Compressive Strength for Four Height-Width Ratios, psi | | | | Average, All Height-Width Ratios |
|---|---|---|---|---|---|---|
| | | 2.00 | 1.00 | 0.75 | 0.50 | |
| VERMICULATE CONCRETE TESTED AIR DRY AT AGE 12 DAYS AFTER 5 DAY CURE | | | | | | |
| 1:4 | 2 by 2 | 398 | 403 | 415<br>403 | 400 | 404 |
| 1:4 | 3 by 3 | 340 | 328 | 343 | 354<br>338 | 341 |
| 1:6 | 2 by 2 | 167 | 175 | 175<br>187 | 169 | 175 |
| 1:6 | 3 by 3 | 157 | 138 | 156 | 162<br>159 | 154 |
| VERMICULATE CONCRETE TESTED MOIST AFTER 7 DAY CURE | | | | | | |
| 1:4 | 2 by 2 | 163 | 163 | 148<br>170 | 167 | 162 |
| 1:4 | 3 by 3 | 130 | 127 | 144 | 142<br>126 | 134 |
| 1:6 | 2 by 2 | 144 | 139 | 151<br>158 | 156 | 150 |
| 1:6 | 3 by 3 | 125 | 123 | 117 | 118<br>124 | 121 |
| Average 1:4 mixes | | 258 | 255 | 271 | 255 | 260 |
| Average 1:6 mixes | | 148 | 144 | 157 | 148 | 150 |

The averages of 8 tests groups each, compiled separately for vermiculite and perlite, showed the type of aggregate did not have a measurable effect on the results:

COMBINED INDICATIONS OF AIR DRY AND MOIST SPECIMENS FOR EACH AGGREGATE—RELATIVE STRENGTH BASIS.

| Height-Width Ratio | 2.00 | 1.00 | 0.75 | 0.50 |
|---|---|---|---|---|
| Average 8 Perlite Test Groups | 95 | 100.5 | 100 | 103 |
| Average 8 Vermiculite Test Groups | 99 | 97 | 102 | 100.5 |

Since the data on height-width effect for the two aggregates were similari it seemed preferable to present the relative strength values for each test round separately.

Table IX, for the specimens of first round, contains the results for height-width ratio specimens which were tested in the air dry condition. It should be explained that the perlite concrete prisms were tested at 9 days because of the fact they had dried to a reasonably uniform moisture condition at this age. On the

## TABLE IX.—RELATIVE AIR DRY STRENGTHS OF INDIVIDUAL SPECIMENS OF 2 SIZES.

Type III cement insulating concretes with different aggregates. Average compressive strength for all height-width ratios on each line equals 100.

| Mix Proportions | Cross-Section, in. | Relative Strength for Four Height-Width Ratios | | | | Average Compressive Strength, psi |
|---|---|---|---|---|---|---|
| | | 2.00 | 1.00 | 0.75 | 0.50 | |
| Expanded Perlite Concrete Air Dry at age 9 days | | | | | | |
| 1:4 | 2 by 2 | 88 | 104 | 106 (2) | 97 | 604 |
| 1:6 | 2 by 2 | 83 | 115 | 93 | 105 (2) | 214 |
| 1:4 | 3 by 3 | 96 | 88 | 107 | 105 (2) | 556 |
| 1:6 | 3 by 3 | 96 | 85 | 107 | 107 (2) | 182 |
| Vermiculite Concrete Air Dry at age 12 days | | | | | | |
| 1:4 | 2 by 2 | 99 | 100 | 101 (2) | 99 | 404 |
| 1:6 | 2 by 2 | 95 | 100 | 103 (2) | 96 | 175 |
| 1:4 | 3 by 3 | 100 | 96 | 101 | 102 (2) | 341 |
| 1:6 | 3 by 3 | 102 | 90 | 103 | 104 (2) | 154 |
| Average all mixes, air-dry test basis | | 95 | 97 | 103 | 103 | |

## TABLE X.—RELATIVE WET STRENGTHS OF INDIVIDUAL SPECIMENS OF 2 SIZES.

Type III cement insulating concretes with different aggregates. Average compressive strength for all height-width ratios on each line equals 100.

| Mix Proportions | Cross-Section, in. | Relative Strength for Four Height-Width Ratios | | | | Average Compressive Strength, psi |
|---|---|---|---|---|---|---|
| | | 2.00 | 1.00 | 0.75 | 0.50 | |
| Expanded Perlite Concrete Moist Cured 7 days | | | | | | |
| 1:4 | 2 by 2 | 95 | 100 | 102 (2) | 100 | 554 |
| 1:6 | 2 by 2 | 99 | 105 | 94 (2) | 109 | 160 |
| 1:4 | 3 by 3 | 103 | 107 | 96 | 97 (2) | 406 |
| 1:6 | 3 by 3 | 100 | 100 | 98 | 100 (2) | 141 |
| Vermiculite Concrete Moist Cured 7 days | | | | | | |
| 1:4 | 2 by 2 | 101 | 101 | 98 (2) | 103 | 162 |
| 1:6 | 2 by 2 | 96 | 93 | 103 (2) | 96 | 150 |
| 1:4 | 3 by 3 | 97 | 95 | 108 | 100 (2) | 134 |
| 1:6 | 3 by 3 | 103 | 102 | 97 | 100 (2) | 121 |
| Average all mixes, moist test basis | | 99 | 100 | 99 | 100 | |

other hand the vermiculite concrete prisms showed too great a range of air dry density at age 9 days making it necessary to allow 3 days additional drying to obtain a more uniform moisture condition at time of test. It is probable that perlite concrete strength for height-widths of 2.00 and 1.00 tended to be lowered slightly because of relatively slower drying of the specimen.

Oven drying would probably provide a more uniform condition for making strength tests on a dried basis but the results would be unrealistic since the concrete is not oven dried in service.

Table X, for specimens of the second round, includes the results for height-width ratio effect when prism specimens were tested moist. The slices were cut from the molded prisms at age 4 days and the sawed faces were capped with sulfur mix on the day of test. As previously mentioned the density and strength level of the 1:4 vermiculite mix were lower in round II because $2\frac{1}{2}$ oz of air entraining agent was added to im-

TABLE XI.—COMPRESSIVE STRENGTH TEST DATA OF INDIVIDUAL SPECIMENS, ROUND I.

Type III cement insulating concretes with air-entraining admixture. Various types of specimens tested air dry after 5 day moist cure.

| | Batch No. 1 | Batch No. 2 | Batch No. 3 | Batch No. 4 |
|---|---|---|---|---|
| Aggregate Type | Perlite | Perlite | Vermiculite | Vermiculite |
| Volume proportions | 1:4 | 1:6 | 1:4 | 1:6 |
| Age at test | 9 days | 9 days | 12 days | 12 days |
| UNIT COMPRESSIVE STRENGTH, PSI | | | | |
| 2 in. cubes | 566<br>534<br>590 | 169<br>184<br>159 | 426<br>411<br>426 | 181<br>210<br>206 |
| Average | 563 | 171 | 421 | 199 |
| 2 by 4 in. cylinders | 528<br>496 | 181<br>188 | 356<br>344 | 159<br>178 |
| Average | 512 | 185 | 350 | 168 |
| 3 by 6 in. cylinders | 519<br>544 | 174<br>131 | 330<br>350 | 128<br>125 |
| Average | 532 | 153 | 340 | 127 |
| 3 by 6-in. paper, air dried | 590 | 151 | 328 (9d) | 105 (9d) |
| oven dried | 620 | 142 | 390 (9d) | 145 (9d) |
| Average all height-widths 2 by 2 prism | 604 | 214 | 404 | 175 |
| Average all height-widths 3 by 3 prism | 556 | 182 | 341 | 154 |
| AVERAGE COMPUTED AIR DRY DENSITY, LB PER CU FT | | | | |
| 2 in. cubes | 38.7 | 25.4 | 35.8 | 27.3 |
| 2 by 4 in. cylinders | 38.2 | 24.8 | 35.7 | 27.5 |
| 3 by 6 in. cylinders | 39.1 | 27.1 | 38.8 | 28.9 |
| 3 by 6 in. paper, air dried | 40.7 | 28.3 | 42.4 | 33.8 |
| oven dried | 35.0 | 24.1 | 34.0 | 25.8 |
| 2 by 2 in. prism slices | 39.4 | 26.4 | 36.7 | 26.8 |
| 3 by 3 in. prism slices | 38.7 | 26.0 | 36.5 | 26.6 |

TABLE XII.—COMPRESSIVE STRENGTH TEST DATA OF INDIVIDUAL SPECIMENS, ROUND II.

Type III cement insulating concretes with air-entraining admixture. Various types of specimens tested wet after 7 day moist cure.

| | Batch No. 5 | Batch No. 6 | Batch No. 7 | Batch No. 8 |
|---|---|---|---|---|
| Aggregate Type | Perlite | Perlite | Vermiculite | Vermiculite |
| Volume proportions | 1:4 | 1:6 | 1:4 | 1:6 |
| Age at test | 7 days | 7 days | 7 days | 7 days |
| UNIT COMPRESSIVE STRENGTH, PSI | | | | |
| 2 in. cubes | 475 | 152 | 152 | 137 |
| | 446 | 154 | 152 | 147 |
| | 471 | 154 | 137 | 132 |
| Average | 464 | 153 | 147 | 139 |
| 2 by 4 in. cylinders | 417 | 146 | 137 | 111 |
| | 497 | 124 | 134 | 121 |
| Average | 457 | 135 | 135 | 116 |
| 2 by 4 in., upper portion | 433 | 121 | 102 | ... |
| lower portion | 436 | 121 | 134 | 143 |
| 3 by 6 in. cylinders | 444 | 90 | 128 | 142 |
| | 436 | 91 | 121 | 140 |
| Average | 440 | 91 | 125 | 141 |
| 3 by 6 in., top portion | 553 | 130 | 111 | 128 |
| middle portion | 453 | 125 | 115 | 155 |
| bottom portion | 393 | 97 | 135 | 145 |
| 3 by 6 in. paper, air dried | 487 | 155 | 151 | 162 |
| oven dried | 527 | 167 | 199 | 229 |
| Average all height-widths 2 by 2 prism | 554 | 160 | 162 | 150 |
| Average all height-widths 3 by 3 prism | 406 | 141 | 134 | 121 |
| AVERAGE COMPUTED DENSITY, AS TESTED, LB PER CU FT | | | | |
| 2 in. cubes | 45.8 | 35.1 | 45.6 | 47.6 |
| 2 by 4 in. cylinders | 46.1 | 34.9 | 45.1 | 47.3 |
| 3 by 6 in. cylinders | 45.9 | 34.7 | 45.8 | 50.0 |
| 2 by 2 in. prism slices | 47.5 | 35.5 | 46.9 | 48.6 |
| 3 by 3 in. prism slices | 45.8 | 35.5 | 46.0 | 48.2 |
| 3 by 6 in. paper, air dried | 39.8 | 27.2 | 32.2 | 35.8 |
| oven dried | 34.4 | 24.2 | 27.5 | 28.8 |

prove placeability. This increased amount of agent reduced the fresh density from 54 to 44 lb per cu ft.

The average results of Tables IX and X show that height-width ratio had a negligible effect in the range of ratios from 2.0 to 0.5. This rule appears valid for air dry and moist specimens in the low density range of 25 to 50 lb per cu ft.

*Evaluation of Various Sizes of Strength Test Specimens:*

A few remarks on our experiences in making different sized specimens may be of interest. The ½ by 1 by 6 in. rectangular tamper specified in ASTM Method

TABLE XIII.—AVERAGE COMPRESSIVE STRENGTH TEST RESULTS FOR VARIOUS SMALL SPECIMENS.

Type III cement insulating concretes with air-entraining admixture. Round I air dry basis: Round II undried basis.

| Volume Proportions<br>Aggregate Type | 1:4<br>Perlite | 1:6<br>Perlite | 1:4<br>Vermiculite | 1:6<br>Vermiculite | Average<br>Level |
|---|---|---|---|---|---|
| **Compressive Strength Air Dry Basis, psi** | | | | | |
| Round I Batch | No. 1 | No. 2 | No. 3 | No. 4 | |
| 2 in. cubes | 563 | 171 | 421 | 199 | ... |
| 2 by 4 cylinders | 512 | 185 | 350 | 168 | ... |
| 3 by 6 cylinders | 532 | 153 | 340 | 127 | ... |
| 4 height-width ratios, 2 by 2 prism | 604 | 214 | 404 | 175 | ... |
| 3 by 3 prism | 556 | 182 | 341 | 154 | ... |
| Average, all specimens | 553 | 181 | 371 | 165 | ... |
| **Compressive Strength Wet Basis, psi** | | | | | |
| Round II batch | No. 5 | No. 6 | No. 7 | No. 8 | |
| 2 in. cubes | 464 | 153 | 147 | 139 | ... |
| 2 by 4 cylinders | 457 | 135 | 135 | 116 | ... |
| 3 by 6 cylinders | 440 | 91 | 125 | 141 | ... |
| 4 height-width ratios, 2 by 2 prism | 554 | 160 | 162 | 150 | ... |
| 3 by 3 prism | 406 | 141 | 134 | 121 | ... |
| Average, all specimens | 464 | 136 | 141 | 133 | ... |
| **Relative Strength, Air Dry Specimens, Average for each mix = 100** | | | | | |
| Round I Batch | No. 1 | No. 2 | No. 3 | No. 4 | |
| 2 in. cubes | 102 | 94 | 113 | 121 | 107 |
| 2 by 4 cylinders | 93 | 102 | 94 | 102 | 98 |
| 3 by 6 cylinders | 96 | 85 | 92 | 77 | 88 |
| 4 height-width ratios, 2 by 2 prism | 109 | 118 | 109 | 106 | 110 |
| 3 by 3 prism | 100 | 101 | 92 | 94 | 97 |
| **Relative Strength, Specimens Undried, Average for each mix = 100** | | | | | |
| Round II Batch | No. 5 | No. 6 | No. 7 | No. 8 | |
| 2 in. cubes | 100 | 113 | 105 | 104 | 106 |
| 2 by 4 cylinders | 98 | 99 | 96 | 87 | 95 |
| 3 by 6 cylinders | 95 | 67 | 89 | 106 | 89 |
| 4 height-width ratios, 2 by 2 prism | 119 | 118 | 115 | 112 | 116 |
| 3 by 3 prism | 88 | 104 | 95 | 91 | 94 |

C 109[4] was found more suitable than metal rods for compacting the specimens in these small molds, because of the lighter weight of the material in the tamper. Although there seemed to be a tendency for better consolidation in 2 in. molds compared to 3 in. molds this is not reflected consistently in our computed values of density of hardened specimens, based on the dimensions of the specimen.

The usual procedures were used for making the specimens. In general, molds were filled in two layers with uniform tamping of each layer. Some faulty 2 by 4 in. cylinders in round I were discarded when it was noticed the top layer had not been tamped. Otherwise all specimens which were molded appear in the tables of data.

Although only a few specimens were tested to determine the effect of oven drying, the results were consistent and showed that after 4 days of air drying the strength of perlite insulating concrete was raised about 5 per cent when oven dried, while the vermiculite insulating concrete strength was raised 30 per cent under the same conditions. After a longer period of air drying the strength increase due to oven drying of vermiculite concrete would probably be lower.

Tables XI and XII containing the results of individual specimens. Table XIII contains the average test results for different specimens.

Although the 2 in. cubes usually showed a higher strength than 2 by 4 in. cylinders and 2 by 2 in. prisms were consistently higher in strength than 3 by 3 in. prisms, the differences in computed density of the specimens did not consistently follow the same trends.

Calculated average strength levels listed in Table XIII show that results with 2 by 4 in. cylinders and sawed sections of 3 by 3 in. prisms agreed closely and were nearest to the average of all specimens.

One point of interest is that the cubes were not capped and all other test pieces were capped with sulfur mix on the day of test.

The molding of cubes and prisms was found easier than making small cylinder specimens and some refinements of procedure should be developed to insure that cylinders will be uniform. Some side tests of selected specimens showed poor top to bottom uniformity in molded 3 by 6 in. cylinders.

A study of the averages given in Table XIII will reveal some differences that indicate the mode of consolidation, as related to the dimensions of the mold, has a greater effect on measured compressive strength than the final size or shape of the specimen.

*Acknowledgment:*

Credit is due Mr. A. L. Bowman of our Research Laboratory who carried out these tests and made most of the calculations.

MR. J. S. DIXON[5] (*by letter*).—Cellular concrete made with preformed foam, water, and portland cement with or without suitable light weight aggregate falls in the class of insulating concrete. Oven dry densities may range from 20 to 90 lb per cu ft. When freshly mixed it is very fluid and, in general, any aggregate incorporated will pass a standard No. 4 sieve.

In view of the above, we would concur with the first paragraph of Sherman's Synopsis. Referring to the data presented in the study:

1. In this we concur; however, our test data indicates that good uniformity occurs at a loading rate of 35 psi to 50 psi per sec.

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[4] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 56), 1956 Suppl. to Book of ASTM Standards, Part 3, p. 129.

[5] Vice-president, National Foam System, Inc., West Chester, Pa.

2. We do not experience this variation in density.

3. No comment, as all our test data is based upon a height-width ratio of 2; namely, 6 by 12 in., 3 by 6 in., and 2 by 4 in. cylinders. Mostly 3 by 6 in. cylinders because of the practicability and readily available 3 by 6 in. waxed cardboard cylindrical molds.

4. We concur that proper curing is essential. However, we have not been able to satisfy ourselves as to the optimum conditions and periods, especially for the low density mixes. Immersion is believed to be not acceptable for low densities.

In further reference to size and shape of compressive test specimens, much is to be said in favor of 3 by 6 in. cylinders in that the molds are so readily available to those wishing to make field specimens. Cube molds now available, are high in cost and can hardly be considered field equipment.

A cube can be tested in any position where a cylinder must be tested in the position cast. We believe there is a strength differential between loading parallel and perpendicular to the direction of casting. (Valore ACI)

Laboratory practice is, we believe, to test cubes made in standard cube molds without capping. There would be a strong tendency to cast cubes in improvised molds, thus opening the way to unsatisfactory results where caps were neglected.

Regarding capping material: Our experience has been that more uniform results are obtained with dry caps (clay sulfur) than with gypsum or cement caps. Moisture gain through the use of gypsum or cement caps when not properly controlled adversely affects compressive strength, especially in the lower densities.

Much can also be said in favor of face grinding, but here again the human element, as in the case of wet caps, must be considered. With oven dry specimens clay sulfur caps are to be recommended.

While this paper is confined for the most part to curing and testing specimens, size and shape of specimens, and condition at time of test, there is also a dire need for a standard procedure on molding insulating concrete which, as previously mentioned, has a high fluidity. Also a standard procedure is needed for handling specimens from the time of molding until tested. Low density insulating concrete due to its low strength as compared with dense concrete by necessity requires a different handling technique.

Mr. Sherman is to be complimented on the work which he has done. It is a step in the right direction which should lead to a tentative standard badly needed.

MR. MORTON SHERMAN (*author's closure*).—The conclusions we have expressed concerning the effects of variations in loading rate, height-width ratio and curing methods are essentially concurred in by the discussors.

Mr. Dixon mentions field specimen molds in discussing specimen size and shape. This points up the ever present conflict between field sampling convenience, and specimen reproducibility and accuracy.

Commercially available 3 by 6-in. cardboard cylinder molds do not have a heavy wax coating, water-tight end seams, or a flat, rigid, metal end plate; thus comparable field accuracy could be obtained just as conveniently with a plywood slab mold, where the material would be cut into cube test specimens in the laboratory, using conventional wood cutting equipment.

Much of Mr. Helm's data covers the subject of moisture content and measured strength; our experience indicates that a series of tests made with identical dry density and cement content insulating concretes, would quite probably indicate the same compressive strength at the same moisture content.

Mr. Helms, the chairman of Subcommittee IIIa on Methods of Testing Concrete for Strength, of ASTM Committee C-9 on Concrete and Concrete Products, is to be particularly thanked for releasing this large quantity of test data for inclusion as a discussion of this paper.

# STUDIES OF FLEXURAL STRENGTH OF CONCRETE—PART 3: EFFECTS OF VARIATIONS IN TESTING PROCEDURES*, [1]

By Stanton Walker and Delmar L. Bloem[2]

## Synopsis

The indicated flexural strength of concrete is more significantly affected by relatively obscure factors than is commonly realized. The investigation described here provides data on modulus of rupture for: different positions of beams as molded; five methods of applying load; beams of different cross-sections; sawed *versus* molded beams; and different moisture conditions of specimens and ages at test. Data are also presented on reproducibility of duplicate tests on the same beam and comparisons between flexural and compressive strength. The data point to the need for a review of current testing procedures. They suggest also that, while the flexural strength test is a useful tool in research and in the laboratory evaluation of concrete ingredients and proportions, it is too sensitive to testing variations to be usable as a basis for the acceptance or rejection of concrete in the field.

It is well known that measured flexural strength is influenced by many factors which are not readily controlled. This fact has become of greater significance in recent years because of the increasing use of modulus of rupture as a basis for the design and acceptance of concrete.

The need for further examination of the reliability of the flexural strength test led to the conduct of a comprehensive investigation at the University of Maryland in the laboratory sponsored jointly by the National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn. This paper covers the third part of that investigation and is devoted principally to conditions of test. The first two parts, dealing with effects of different aggregates, cements and curing conditions, have been published elsewhere (**1**,**2**).[3] Other reports are to follow.

The data reported here are from several different investigations. Each is outlined and discussed separately.

## Studies of Testing Procedures

One objective was to investigate the testing procedures specified in the ASTM Method of Test for Flexural Strength of Concrete (C 78 - 49).[4] That method requires, among other things, that the test specimen shall be turned on its side when loaded. It presents a diagram of a loading apparatus which appears to have certain deficiencies. It requires capping of surfaces in contact with the bearings if they are not plane within 0.002 in., as stipulated for compression specimens.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] For references to Parts 1 and 2 see list of references appended to this paper (**1**, **2**).

[2] Director and Associate Director of Engineering, respectively, of the National Sand and Gravel Assn., and the National Ready Mixed Concrete Assn. Washington, D. C.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1138.

[4] Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 - 49), 1955 Book of ASTM Standards, Part 3, p. 1323.

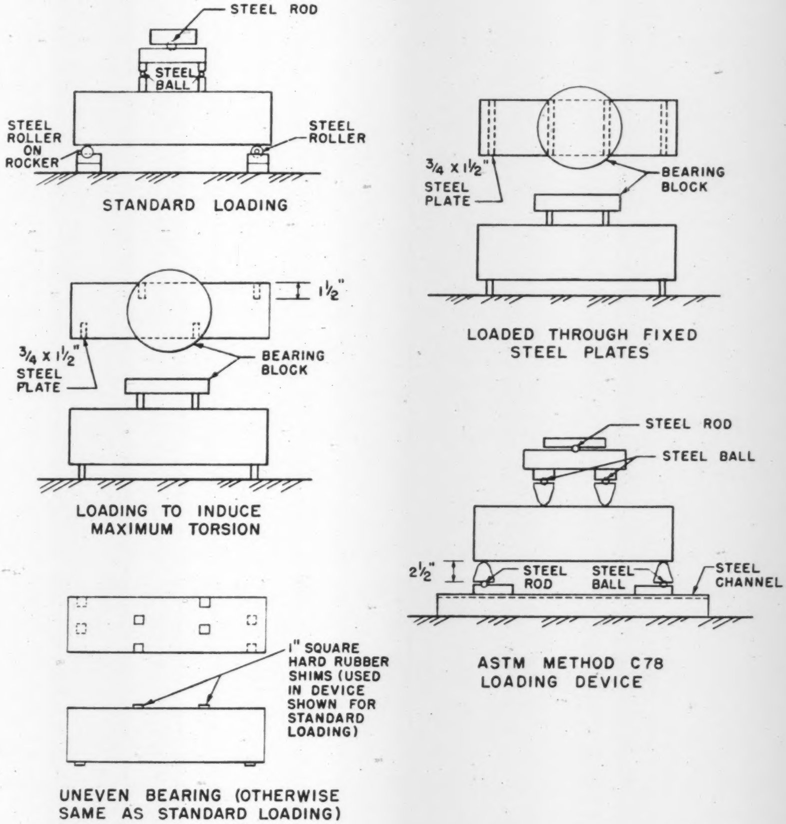
STEEL ROD
STEEL BALL
STEEL ROLLER ON ROCKER
STEEL ROLLER
STANDARD LOADING
3/4 x 1 1/2" STEEL PLATE
BEARING BLOCK
LOADED THROUGH FIXED STEEL PLATES
1 1/2"
3/4 x 1 1/2" STEEL PLATE
BEARING BLOCK
LOADING TO INDUCE MAXIMUM TORSION
STEEL ROD
STEEL BALL
2 1/2"
STEEL ROD
STEEL BALL
STEEL CHANNEL
ASTM METHOD C78 LOADING DEVICE
1" SQUARE HARD RUBBER SHIMS (USED IN DEVICE SHOWN FOR STANDARD LOADING)
UNEVEN BEARING (OTHERWISE SAME AS STANDARD LOADING)

FIG. 1.—Schematic Diagram of Loading Methods (Series J 102).

In a study of these factors, 7 different loading conditions were used, as shown in Fig. 1 and described below:

(*a*) For conditions 1, 2, and 3, the specimens were oriented differently in relation to their position as molded, as follows: (1) side in tension, (2) top in tension, and (3) bottom in tension. The loading device was that regularly used in our laboratory, complying with all basic requirements of Method C 78[4] but not with the diagram shown there.

(*b*) For conditions 4, 5, 6, and 7, beams were loaded with the side in tension, using 4 additional methods of transmitting the load as follows: (4) through fixed steel plates, (5) through fixed blocks in such a way as to induce maximum torsion, (6) through shims in the regular device to simulate application of load to an irregular surface, and (7) in apparatus built strictly in accordance with the diagram in C 78.[4]

Conditions 4 through 7 should be compared with condition 1 as standard. For conditions 2 and 3, bearing surfaces at the top of the beam as molded were capped with neat cement. Other surfaces, cast against the steel molds, were checked and found to be satisfactorily plane.

In this portion of the investigation, beams were 6 by 6 by 21 in. The concrete was made with quartz sand and gravel, well graded up to $\frac{3}{4}$ in. Both normal and air-entrained concretes were tested. The concrete was designed to contain 6 sacks of cement per cubic yard with a slump of 2 to 3 in. and a $b/b_o$ of 0.61 (ratio of volume of coarse aggregate, on a dry rodded

TABLE I.—EFFECT OF LOADING CONDITIONS ON LEVEL AND UNIFORMITY OF FLEXURAL STRENGTH TESTS (SERIES J-102).

For all concrete, coarse aggregate was a natural quartz gravel graded 35 per cent ¾ to ½ in., 30 per cent ½ to ⅜ in. and 35 per cent ⅜ to No. 4; fine aggregate was natural siliceous sand with a fineness modulus 2.77; $b/b_0 = 0.61$.
For non-air-entraining concrete: sand 44.6 per cent of total aggregate, average cement factor 5.95 sacks per cu yd, water 35.7 gal per cu yd, *w/c* 6.00 gal per sack, slump 2.4 in., and air 2.0 per cent.
For air-entraining concrete: sand 43.0 per cent of total aggregate, average cement factor 6.01 sacks per cu yd, water 33.4 gal per cu yd, *w/c* 5.55 gal per sack, slump 2.5 in., and air 4.3 per cent.
For description of loading conditions, see text and Fig. 1.

| Batch | Modulus of Rupture, psi | | | | | | |
|---|---|---|---|---|---|---|---|
| | Standard Loading Device with Tension in | | | Fixed Plate Reactions | Maximum Induced Torsion | Uneven Bearing | ASTM C 78 Device |
| | Side (1) | Top (2) | Bottom (3) | (4) | (5) | (6) | (7) |
| | | | NON-AIR-ENTRAINING CONCRETE | | | | |
| A | 672 | 630 | 633 | 685 | 587 | 608 | 589 |
| B | 650 | 602 | 627 | 663 | 522 | 633 | 631 |
| C | 624 | 677 | 656 | 579 | 530 | 611 | 514 |
| D | 612 | 649 | 626 | 673 | 528 | 649 | 601 |
| E | 619 | 627 | 670 | 590 | 593 | 613 | 542 |
| Average | 635 | 637 | 642 | 638 | 552 | 623 | 575 |
| Per cent[a] | 100 | 100 | 101 | 100 | 87 | 98 | 91 |
| Coefficient Variation, per cent[b] | 3.9 | 4.4 | 3.0 | 7.8 | 6.3 | 2.8 | 8.2 |
| | | | AIR-ENTRAINING CONCRETE | | | | |
| A | 620 | 669 | 658 | 633 | 607 | 648 | 633 |
| B | 631 | 651 | 606 | 576 | 522 | 640 | 587 |
| C | 587 | 618 | 650 | 633 | 596 | 585 | 574 |
| D | 590 | 627 | 580 | 629 | 573 | 646 | [c] |
| E | 634 | 612 | 617 | 576 | 581 | 572 | 566 |
| Average | 612 | 635 | 622 | 610 | 576 | 618 | 590 |
| Per cent[a] | 100 | 104 | 102 | 100 | 94 | 101 | 96 |
| Coefficient Variation, per cent[b] | 3.7 | 3.8 | 5.2 | 5.0 | 6.3 | 5.9 | 5.1 |

[a] Strengths for different loading conditions expressed as percentages of strength for Method 1.

[b] Coefficient of variation $= \sqrt{\frac{\Sigma(x - \bar{x})^2}{n - 1}} \frac{(100)}{\bar{x}}$ where $x$ = individual strength, $\bar{x}$ = average strength and $n$ = number of tests.

[c] Specimen destroyed in handling.

basis, to the volume of concrete). It was mixed in a $3\frac{1}{2}$ cu ft mixer, and one beam for each of the 7 loading conditions was made from each batch. Five batches of each type concrete were tested to provide reliable average data. The results are summarized in Table I and Fig. 2.

*Position of Beam:*

The first three columns of Table I and the first three bars of Fig. 2 show the effects of orientation of the beams—that is, side, top, or bottom in tension—in the standard apparatus. It is evident that the position of the specimen in relation to its position as molded had no significant effect on the modulus of rupture. Also, the values of coefficient of variation, shown in Table I for the three loading positions, reveal no significant differences in reproducibility. Analyses of variance made by J. B. Blackburn, Associate Professor of Civil Engineering at the University of Maryland, confirm the lack of significance of differences due to specimen orientation (3).

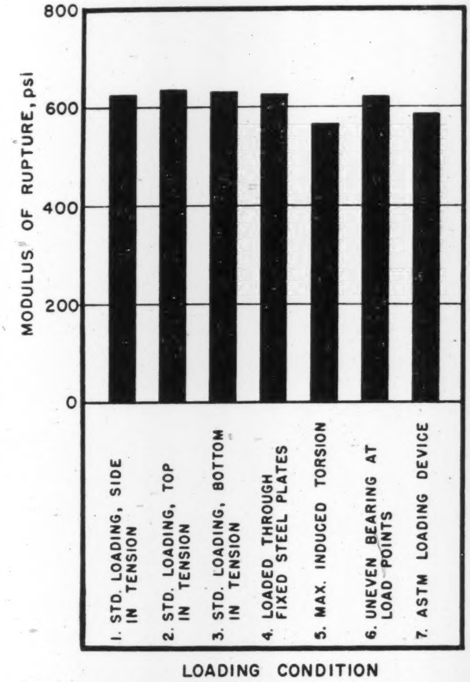

Fig. 2.—Effect of Loading Condition on Measured Flexural Strength (Series J 102).

*Methods of Transmitting Load:*

Comparisons between condition 1 and conditions 4 through 7 in Table I and Fig. 2 indicate the significance of variations in loading method.

Condition 4 where the bearings for both loading and support were simple rectangular steel plates with no provision for movement, gave measured strengths equal to those secured in the regular loading device. While this might not have been true for less accurately molded specimens, it does suggest that freedom from restraint may not be as essential as is generally supposed. It should be noted, however, that the coefficient of variation tended to be slightly higher for the fixed plates than for standard loading. Thus, failure to provide unrestrained loading may reduce uniformity of test results, even though it does not affect the average strength level.

Condition 5 seriously violated the principles of flexure testing outlined in C 78. Not only were the bearings rectangular plates with no provision for movement, but the load was applied in such a manner as to induce maximum torsion. While the measured flexural strength was reduced significantly, the reduction was, nevertheless, less than seems consistent with the magnitude of the violation of accepted testing procedure. For the non-air-entrained concrete, the strength was reduced 13 per cent below that for standard loading and, for air-entrained concrete, 6 per cent. Uniformity of test results was decreased as indicated by the higher coefficients of variation shown in Table I for condition 5.

Condition 6 represented an attempt to simulate the condition of bearings in

### TABLE II.—EFFECT OF DEPTH OF SPECIMEN ON MEASURED FLEXURAL STRENGTH (SERIES 155C).

Non-air-entraining concrete made with quartz gravel and siliceous sand designed to contain 6 sacks of cement per cu yd with slump of 2 to 3 in.

| Batch | Cement, sacks per cu yd | Water | | Slump, in. | Unit Weight, lb per cu ft | Per cent, Air | 28-day Modulus of Rupture, psi, for Different Beam Cross-Sections, in. | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Gal per cu yd | Gal per sack | | | | 3 × 4 | 6 × 6 | 8 × 8 | 10 × 10 |
| 1-IN. MAX SIZE COARSE AGGREGATE; $b/b_0$ = 0.72; SAND 33.5 PER CENT TOTAL ABSOLUTE VOLUME AGGREGATE | | | | | | | | | | |
| A | 6.01 | 31.5 | 5.24 | 2.6 | 149.6 | 1.2 | 712 | 693 | 620 | 603 |
| B | 6.00 | 31.1 | 5.18 | 2.2 | 149.6 | 1.5 | 710 | 659 | 614 | 623 |
| C | 6.00 | 31.6 | 5.28 | 2.6 | 149.6 | 1.1 | 680 | 688 | 643 | 662 |
| D | 5.99 | 32.1 | 5.36 | 2.7 | 149.5 | 1.1 | 720 | 649 | 620 | 620 |
| E | 6.01 | 32.4 | 5.38 | 2.8 | 149.3 | 0.7 | 643 | 621 | 599 | 634 |
| F | 6.00 | 31.9 | 5.31 | 2.8 | 149.5 | 1.2 | 685 | 633 | 639 | 614 |
| G | 6.03 | 31.4 | 5.21 | 1.9 | 150.1 | 1.0 | 655 | 638 | 625 | 631 |
| H | 6.03 | 31.7 | 5.26 | 2.0 | 150.4 | 0.7 | 645 | 675 | 607 | 609 |
| K | 6.02 | 31.9 | 5.30 | 2.2 | 150.0 | 0.8 | 676 | 643 | 597 | 608 |
| L | 6.00 | 32.0 | 5.33 | 2.6 | 150.1 | 1.0 | 685 | 634 | 611 | 612 |
| Average | 6.01 | 31.8 | 5.28 | 2.4 | 149.8 | 1.0 | 681 | 653 | 618 | 622 |
| Standard Deviation[a] | | | | | | | 27 | 24 | 16 | 17 |
| $v$, per cent[b] | | | | | | | 4.0 | 3.7 | 2.6 | 2.7 |
| 2-IN. MAX SIZE COARSE AGGREGATE; $b/b_0$ = 0.80; SAND 29.8 PER CENT TOTAL ABSOLUTE VOLUME AGGREGATE | | | | | | | | | | |
| A | 5.99 | 29.0 | 4.84 | 1.5 | 151.0 | 1.1 | 746 | 653 | 602 | 569 |
| B | 5.98 | 30.0 | 5.02 | 2.4 | 151.2 | 0.7 | 690 | 631 | 577 | 518 |
| C | 5.98 | 29.9 | 5.00 | 2.8 | 151.2 | 0.7 | 677 | 659 | 571 | 546 |
| D | 5.97 | 30.2 | 5.06 | 2.4 | 151.0 | 0.8 | 625 | 612 | 540 | 570 |
| E | 6.00 | 29.7 | 4.95 | 2.3 | 151.3 | 0.7 | 597 | 619 | 639 | 581 |
| F | 5.96 | 30.0 | 5.03 | 2.5 | 150.5 | 1.1 | 758 | 623 | 661 | 527 |
| G | 5.98 | 30.0 | 5.02 | 2.8 | 151.2 | 0.7 | 748 | 630 | 593 | 488 |
| H | 6.02 | 29.4 | 4.88 | 1.5 | 151.6 | 0.7 | 716 | 656 | 659 | 552 |
| K | 6.00 | 30.7 | 5.12 | 3.1 | 151.7 | 0.2 | 631 | 656 | 605 | 552 |
| L | 6.00 | 30.2 | 5.04 | 2.8 | 152.0 | 0.4 | 635 | 581 | 596 | 542 |
| Average | 5.99 | 29.9 | 5.00 | 2.4 | 151.3 | 0.7 | 682 | 632 | 604 | 545 |
| Standard Deviation[a] | | | | | | | 59 | 25 | 39 | 27 |
| $v$, per cent[b] | | | | | | | 8.2 | 4.0 | 6.5 | 5.0 |

[a] Standard deviation of individual test results, $\sigma = \sqrt{\frac{\Sigma(x - \bar{x})^2}{n - 1}}$ where $x$ = individual strength, $\bar{x}$ = average strength, and $n$ = number of tests, in this case 10.

[b] Coefficient of variation, $v$ = standard deviation expressed as per cent of average.

contact with an irregular surface. Shims were inserted at the bearing points. Otherwise the apparatus was standard, with freedom of movement at all bearings. The measured flexural strengths were essentially the same as for the standard method. The average of the coefficients of variation for the normal and the air-entrained concrete also compared favorably with those for the standard method.

For condition 7, the loading device

diagramed in Fig. 1 of C 78[4] was used. This apparatus is unstable. Even with meticulous positioning of the specimen and of the bearings, the best condition that can be secured is one of unstable equilibrium. In order to use the apparatus, it was necessary to place shims under the rocker-type lower supports to prevent them from tilting. These shims were placed inside the middle third in order that the bearings could move with elongation of the bottom surface of the beam. The strengths secured for this apparatus were significantly lower than for the one which was considered standard, and the coefficient of variation, particularly for the non-air-entrained concrete, was high.

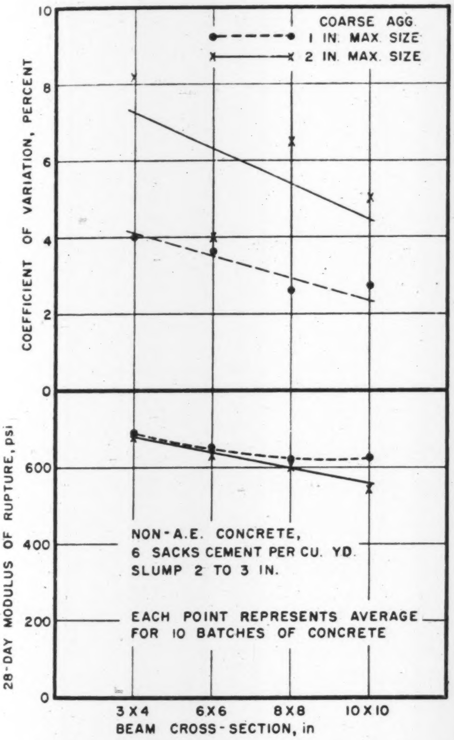

FIG. 3.—Effect of Depth of Specimen on Level and Reproducibility of Measured Flexural Strength (Series 155 C).

The rocker-type lower supports were undoubtedly the cause of the reduced measured strength. Any outward tilting of these supports, due either to initial deviation from the vertical or to elongation of bottom fibers during test, would create a longitudinal component which would produce tension in the lower fibers and reduce the load-carrying capacity of the beam.

It should not be inferred from the results for conditions 1 through 6 that attention to loading procedure can be neglected. A proper loading device, reasonably free of restraint, is essential. On the other hand, meticulous attention to planeness of surface to insure uniform bearing at load and reaction points may not be highly critical. A loading device with cylindrical lower supports, free to roll in the direction of the axis of the beam, with one of them free to rock transversely, seems to represent a preferred type of bearing. Rocker supports which tend to be unstable can produce longitudinal stresses and should not be used.

## CROSS-SECTION OF SPECIMEN

Method C 78[4] refers to the ASTM Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192)[5] and the ASTM Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31).[6] These require the minimum cross-sectional dimension of the test specimen to be at least three times the maximum nominal size of the coarse aggregate. That means that the commonly-used 6 by 6-in. beam is applicable only to concrete made with aggregates 2 in. or less in size. Also, as shown both by theory (4,5) and by laboratory tests (6,7), modulus of rupture varies with depth of specimen—width and length having no substantial effect (6,8).

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[5] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 55), 1955 Book of ASTM Standards, Part 3, p. 1318.

[6] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 – 55), 1955 Book of ASTM Standards, Part 3, p. 1314.

In a study of the significance of specimen size, tests were made of beams having dimensions of: 3 by 4 by 16 in., 6 by 6 by 21 in., 8 by 8 by 27 in., and 10 by 10 by 33 in. Two classes of concrete were made with quartz sand and gravel well graded, in one case, up to 1 in. and, in the other, up to 2 in. The concrete was non-air-entrained. It was designed to contain 6 sacks of cement per cu yd with a slump of 2 to 3 in. and a $b/b_o$ of 0.72 for the 1-in. maximum size and 0.80 for the 2-in. maximum size. For each class of concrete, one beam of each size was molded from each batch. Ten batches were made and tested for each class. The specimens were loaded at the third-points over spans of 15 in. for the 4-in. deep beams and 18, 24, and 30 in. for the 6, 8, and 10-in. depths, respectively.

The data are summarized in Table II and Fig. 3. Reductions in measured modulus of rupture with increasing depth of specimen were, in general, consistent with those reported by other investigators (5,6,7). For the 2-in. aggregate, the relationship was approximately a straight line indicating a reduction of about 45 psi or 7 per cent for each increase of 2 in. in depth. In the case of the 1-in. aggregate, measured flexural strength was reduced as depth was increased from 4 to 8 in., but there was no further reduction between 8 and 10 in. The reason for this inconsistency is not apparent. Between the 4 and 8-in. depths, the modulus of rupture decreased at the rate of about 30 psi or 5 per cent for each 2-in. increase in depth.

As shown in the upper portion of Fig. 3, the uniformity of the test results tended to improve as depth of beam increased. The relationship is marred by what appears to be an inconsistently low coefficient of variation for the 6-in. beams made with 2-in. aggregate, but the trend seems evident. It is reasonable to suppose that the improved uniformity was related to the increasing ratio of specimen

TABLE III.—COMPARISONS BETWEEN MOLDED AND SAWED BEAMS (SERIES 155E).

Concrete made with quartz gravel graded from 1½ in. to No. 4 and siliceous sand having fineness modulus of 2.7. Average cement factor 5.93 sacks per cu yd, water 30.0 gal per cu yd, w/c 5.05 gal per sack, slump 2.7 in. and air 5.1 per cent.

| Batch | Moist-Cured Specimens[a] | | Specimens with Drying Interval[b] | |
|---|---|---|---|---|
| | Molded | Sawed | Molded | Sawed |
| A | 646<br>533<br>489<br>548 | 407<br>430<br>476<br>464 | 558<br>583<br>568<br>618 | 412<br>436<br>338<br>430 |
| | 554 | 444 | 582 | 404 |
| B | 504<br>595<br>534<br>609 | 523<br>337<br>503<br>403 | 554<br>525<br>520<br>464 | 392<br>311<br>429<br>339 |
| | 560 | 442 | 516 | 368 |
| C | 573<br>563<br>590<br>540 | 396<br>462<br>417<br>321 | 524<br>484<br>487<br>458 | 433<br>429<br>397<br>400 |
| | 566 | 399 | 488 | 415 |
| Grand average | 560 | 428 | 529 | 395 |
| Per cent Molded | 100 | 76 | 100 | 75 |
| Standard Deviation[c] | 45 | 60 | 49 | 43 |
| $v$, per cent[d] | 8.0 | 14.0 | 9.3 | 10.9 |

[a] Beams moist-cured continuously until tested at the age of 16 days.

[b] Beams cured 14 days in standard moist room at 73 F, air-dried at 100 F for 7 days, then immersed in water at 73 F for 2 days and tested at the age of 23 days.

[c] Standard deviation $= \sqrt{\frac{\Sigma(x - \bar{x})^2}{n - 1}}$ where $x$ = individual strength, $\bar{x}$ = average and $n$ = number of tests, in this case 12.

[d] Coefficient of variation, $v$ = standard deviation expressed as per cent of average strength.

size to maximum size of aggregate. Any imbalance in distribution of the larger aggregate particles would be expected to become less critical as the cross-section of the beam increased.

It is of interest that, in spite of its lower water-cement ratio, concrete with the larger maximum size aggregate tended to develop slightly lower flexural strengths than that with the smaller aggregate. This is consistent with data reported by Kellerman (7) which showed that, for a constant cement factor and slump, modulus of rupture decreased progressively as maximum size of coarse aggregate was increased from $\frac{3}{4}$ to $2\frac{1}{2}$ in.

Uniformity of flexural strength tests, as indicated by coefficients of variation, was considerably better for the 1-in. than for the 2-in. coarse aggregate. This again is consistent with Kellerman's findings and with the reasoning above regarding the expectation of improvement in uniformity with increasing ratio of specimen size to maximum size of aggregate.

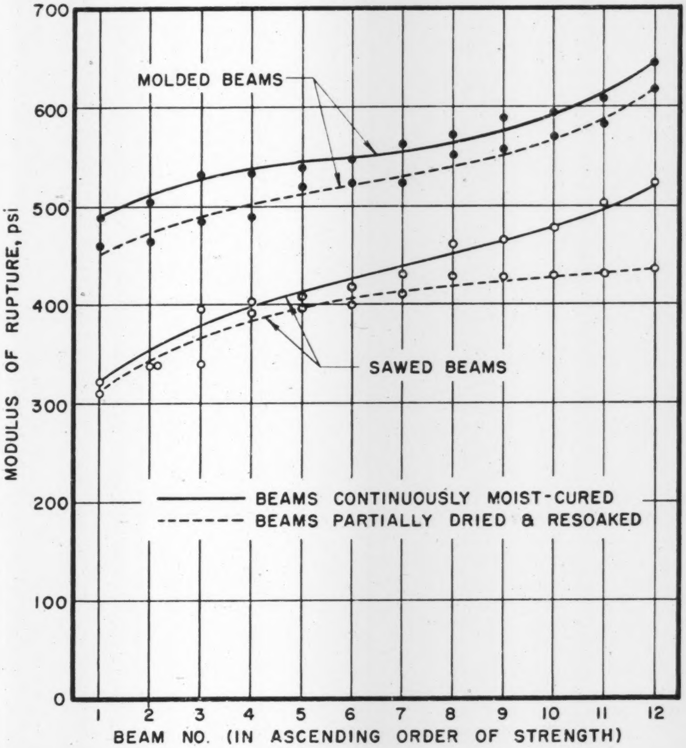

FIG. 4.—Relationship Between Flexural Strengths of Sawed and Molded Beams of Same Concrete (Series 155 E).

## SAWED *Versus* MOLDED BEAMS

Specifications frequently provide for tests of beams sawed from the finished work when molded specimens have failed to produce the strength expected. Some experiences have indicated that, for certain aggregates at least, sawed specimens may give substantially lower strengths than comparable molded specimens (**9,10**).

Tests were made comparing molded 3 by 4 by 16-in. beams with beams of the same dimensions sawed from 6 by 6 by 36-in. specimens molded from the same batches as the smaller ones. The aggregate was quartz sand and gravel, well graded up to 1½ in. The concrete was designed to contain 6 sacks of cement per cubic yard with a slump of 2 to 3 in., an air content of 4.5 per cent and a $b/b_o$ of 0.73. Three rounds were made. Specimens were tested after continuous moist curing and also after moist curing followed by a drying period and then 48 hr of soaking.

From each batch of concrete, a total of 10 beams, eight 3 by 4 by 16-in. and two 6 by 6 by 36-in., were molded. All beams were cured to the age of 14 days in the standard moist room at 73 F. At that time, the large beams were sawed in half transversely, providing two pieces 6 by 6 by 18 in. From one of these halves, two 3 by 4 by 18-in. beams were prepared by sawing 1 in. off each side of the specimen as molded and then cutting the resultant 4 by 6 by 18-in. piece in half along the horizontal center line as molded. The sawed 3 by 4 by 18-in. beams were then immersed in water at 73 F for 48 hr and tested for modulus of rupture, using third-point loading on a 15-in. span with the 4-in. dimension vertical. Four of the 3 by 4 by 16-in. molded beams were also tested at that time (16 days), having been kept continuously moist since molding.

The second 6 by 6 by 18-in. piece of each large beam was placed in air at 100 F immediately after sawing. After 7 days of drying, these were sawed into 3 by 4 by 18-in. beams as described above, immersed in water for 48 hr and tested for modulus of rupture, simulating the standard requirements for specimens removed from hardened concrete.[7] The remaining four 3 by 4 by 16-in. molded beams from each batch were also air-dried at 100 F from the age of 14 to 21 days, soaked 48 hr, and tested for modulus of rupture along with their companion sawed beams.

Results of the tests are shown in Table III and Fig. 4. For the same curing condition, the sawed specimens produced measured moduli of rupture averaging about 25 per cent less than secured with the molded beams. A more realistic comparison (for evaluating the degree to which specimens sawed from concrete in the field might reflect conformance with strength requirements for molded beams) is between moist-cured molded beams and sawed beams which had had an interval of drying. For those conditions, the sawed beams tested lower than the companion molded beams by almost 30 per cent (395 psi as compared with 560 psi).

These tests were confined to a single coarse aggregate. It is not unlikely that concrete with a softer aggregate might have produced sawed beams comparing more favorably with the molded ones. Even though a wet-cutting diamond blade was used, the sawing may in this case have damaged the bond between the hard quartz particles and the mortar.

Another possible cause for the differences in strength may lie in the orientation of coarse aggregate particles with respect to the maximum tensile stresses. In beams with sawed surfaces in tension, the maximum fiber stresses could occur normal to the faces of aggregate particles (where the cut bisects the particle); in molded beams, the maximum stresses must of necessity be tangential to the surface of the particle where it was in contact with the mold.

Regardless of the cause, it is evident that sawed beams may under certain conditions provide an unrealistically low indication of the flexural strength potential of the concrete.

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[7] Methods of Securing, Preparing and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 – 49), 1955 Book of ASTM Standards, Part 3, p. 1360.

## Moisture Content and Age

Both Method C 31[6] and Method C 192[5] require test specimens made to check the adequacy of design to be tested in a moist condition. Researches conducted several years ago at the Bureau of Public Roads (11,12,13) demonstrated the great sensitivity of measured flexural strength to the moisture condition of beams at the time of test. It was noted particularly that the breaking load was sharply reduced by partial drying of saturated specimens. It was also observed (11) that thorough (as opposed to partial) drying produced an increase in measured strength.

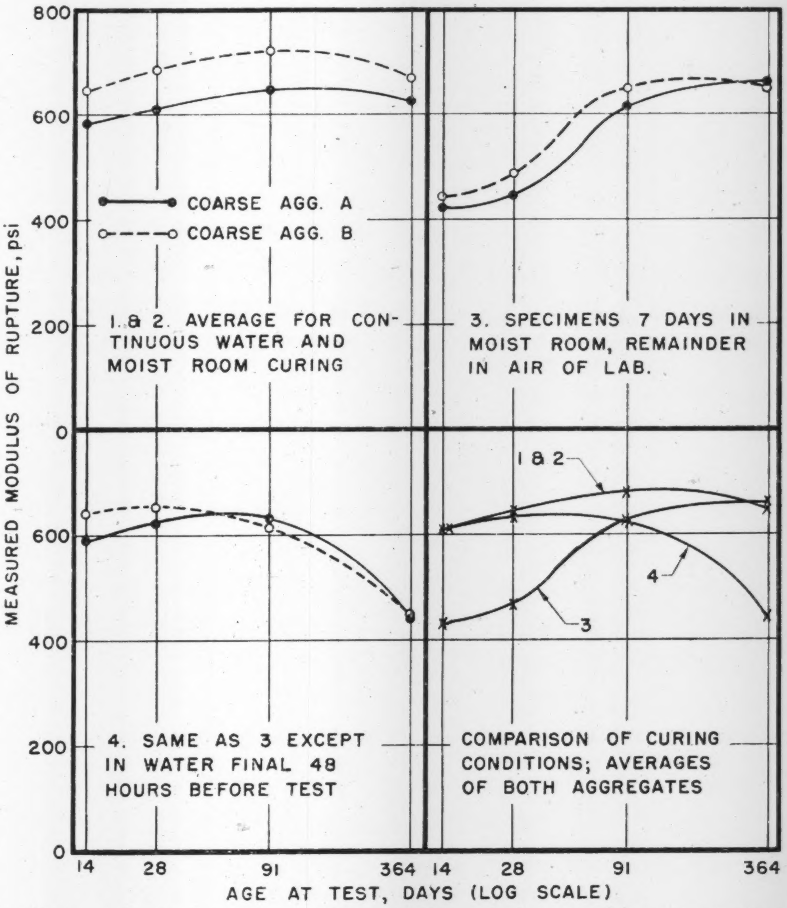

Fig. 5.—Effects of Curing Condition on Measured Flexural Strength of Concrete (Series 159 A).

Tests were made to supplement this earlier work and to secure data more specifically related to problems of testing. In one group (Series 159A), air-entrained concretes of 3 cement factors were made with two coarse aggregates—one of high and one of moderately low flexural strength-producing properties. For each of the 6 types of concrete, tests for modulus of rupture were made at ages of 14, 28, 91, and 364 days after each of 4 methods of curing:

1. Continuously in the standard moist room,
2. Continuously in saturated limewater at 73 F,
3. Continuously in laboratory air after 7 days in the moist room, and
4. Same as No. 3 except that beams were immersed in water for the final 48 hr before test.

Except for the 1-yr tests which have only recently become available, the detailed data from these studies have already been published (2) and are not reproduced here. The principal results are shown graphically in Fig. 5, in which data for the 3 cement factors have been averaged. For the continuously moist-cured specimens, there was a distinct difference in flexural strength between the two coarse aggregates. For specimens which were allowed to dry after 7 days, the difference between aggregates was slight at the earlier ages and completely disappeared at 1 yr. Similarly, for the resoaked specimens, there was little difference between aggregates at 14 or 28 days and none at 3 months and 1 yr.

Permitting the beams to dry after 7 days caused large reductions in strength at early ages. As age increased, the air-dried beams gained considerably in relation to the moist-cured specimens until, at 1 yr, strengths were about equal. Re-

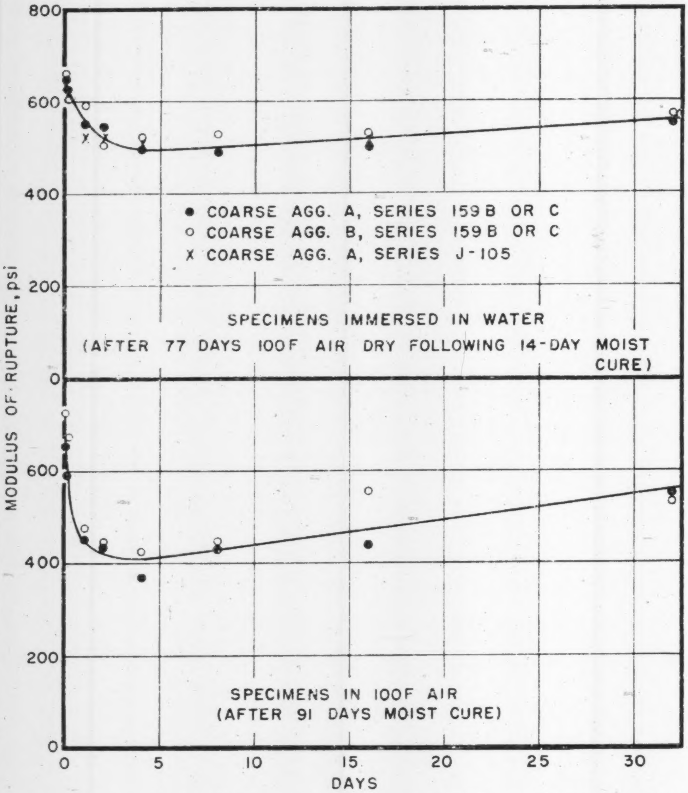

FIG. 6.—Effects of Changes in Moisture Condition on Measured Modulus of Rupture (Series 159 B and C).

soaking increased the measured strength of the dried beams at early ages but reduced it sharply at 1 yr. It can be reasoned that drying of the specimens was detrimental during the early periods, with initial shrinkage of the surface inducing tensile stresses in the outer fibers and reducing load-carrying capacity. As drying became more thorough, the differential shrinkage disappeared and the dry concrete had a sufficient strength advantage (because of being dry) to overcome the more favorable curing of the continuously moist specimens. According to this reasoning, resoaking would tend to relieve the surface tensile stresses at early ages and increase measured strength, but might reduce strength after drying had become uniform. The extremely large strength reduction due to resoaking at 1 yr is not so readily rationalized since the strength fell considerably below the level of comparable tests at earlier ages. It may have been that the very thorough drying followed by immersion in water caused sufficiently disruptive volume changes to reduce the strength.

Further tests on the problem of moisture condition involved the progressive resaturation of thoroughly dried beams (Series 159B) and the progressive drying of continuously moist-cured beams (Series 159C). Concretes were the same as in the first study of curing but only one cement factor was employed. For Series 159B, beams were moist-cured 14 days, dried at 100 F for 77 days and then tested for modulus of rupture after periods of immersion in water ranging from 0 to 32 days. In Series 159C, the beams were moist-cured 91 days and tested after periods of drying at 100 F ranging from 0 to 32 days. Series 159B was repeated with one aggregate (in Series J-105) as a further check on the reliability of the data.

Here again, the detailed data are already available (2). The principal results, pertinent to the subject of this paper, are shown in Fig. 6.

The data confirm the reasoning set forth above concerning the effects of changes in moisture on measured modulus of rupture. Thoroughly dried specimens suffered a sharp reduction in strength upon being immersed in water. The strength reached a minimum after 4 days' immersion and increased slightly thereafter. Weight change measurements indicated that the leveling off of strength was coincident with attainment of an essentially stable moisture content. The later slight strength increase can probably be attributed to the benefit of additional curing, since the specimens had been moist-cured initially for only 14 days.

Progressive drying of thoroughly cured specimens likewise caused an initial drop in strength followed by a moderate increase. Apparently, induced tensile stresses resulted in the decrease, which was later partially offset by the higher strength of the dry concrete as a more uniform moisture content was approached. In this case, specimens were still losing weight after 32 days at 100 F, indicating that complete drying had not been attained.

With regard to the meticulous attention required to secure accurate flexural strength results, it should be noted that, in the latter tests, drying for only 30 min caused an average reduction of 8 per cent in measured modulus of rupture. Thus, the apparently minor lapse of permitting a slight amount of drying to occur before testing could easily result in apparent failure to meet strength requirements.

From these studies, it is evident that flexural strength tests are not only highly sensitive to changes in moisture but also that they provide only a rough indication of load-carrying capacity of a con-

## TABLE IV. REPRODUCIBILITY OF DUPLICATE FLEXURAL STRENGTH TESTS ON SAME BEAM (SERIES 155D).

Air-entraining concretes of 3 cement factors made with each of 4 brands of cement; 5 batches made on different days for each of the 12 conditions; one 6 by 6 by 36 in. beam molded from each batch to provide 2 tests for modulus of rupture at 28 days after standard moist-room curing.
For all conditions, design slump was 2 to 3 in., air content 4.5 ± 0.5 per cent, and $b/b_0$ 0.72; coarse aggregate natural quartz gravel graded from 1½ in. to No. 4.

| Cement Source Number | Batch | Moduli of Rupture, psi, for Different Cement Factors | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 5 Sacks | | | 6 Sacks | | | 7 Sacks | | |
| | | Test 1 | Test 2 | Range | Test 1 | Test 2 | Range | Test 1 | Test 2 | Range |
| 1 | A[a] | 671 | 595 | 76 | 714 | 618 | 96 | 708 | 617 | 91 |
| | B[a] | 648 | 618 | 30 | 684 | 688 | 4 | 683 | 696 | 13 |
| | C | 548 | 559 | 11 | 618 | 628 | 10 | 633 | 665 | 32 |
| | D | 604 | 640 | 36 | 644 | 657 | 13 | 725 | 672 | 53 |
| | E | 519 | 596 | 77 | 629 | 624 | 5 | 634 | 608 | 26 |
| | Average | 598 | 602 | 46 | 659 | 643 | 26 | 677 | 652 | 43 |
| 2 | A[a] | 574 | 562 | 12 | 550 | 625 | 75 | 685 | 697 | 12 |
| | B[a] | 508 | 516 | 8 | 567 | 658 | 91 | 646 | 664 | 18 |
| | C | 609 | 568 | 41 | 651 | 583 | 68 | 625 | 661 | 36 |
| | D | 548 | 578 | 30 | 558 | 573 | 15 | 609 | 662 | 53 |
| | E | 554 | 481 | 73 | 628 | 611 | 17 | 695 | 668 | 27 |
| | Average | 559 | 541 | 33 | 591 | 610 | 53 | 652 | 670 | 29 |
| 3 | A[a] | 538 | 492 | 46 | 578 | 611 | 33 | 614 | 583 | 31 |
| | B[a] | 533 | 512 | 21 | 496 | 554 | 58 | 575 | 483 | 92 |
| | C | 532 | 540 | 8 | 590 | 608 | 18 | 598 | 577 | 21 |
| | D | 534 | 500 | 34 | 566 | 600 | 34 | 612 | 611 | 1 |
| | E | 562 | 508 | 54 | 542 | 547 | 5 | 588 | 579 | 9 |
| | Average | 540 | 510 | 33 | 554 | 584 | 30 | 597 | 567 | 31 |
| 4 | A[a] | 488 | 521 | 33 | 489 | 531 | 42 | 567 | 528 | 39 |
| | B[a] | 495 | 509 | 14 | 555 | 562 | 7 | 597 | 552 | 45 |
| | C | 483 | 513 | 30 | 537 | 602 | 65 | 599 | 595 | 4 |
| | D | 512 | 518 | 6 | 602 | 569 | 33 | 577 | 625 | 48 |
| | E | 480 | 472 | 8 | 577 | 489 | 88 | 608 | 571 | 37 |
| | Average | 492 | 507 | 18 | 552 | 551 | 47 | 590 | 574 | 35 |

Over-all average Test 1 = 588 psi
Over-all average Test 2 = 584 psi
Over-all average Range = 35 psi
Within-specimen standard deviation = 0.8865(35) = 31 psi
Within-specimen coefficient of variation = 31/586 × 100 = 5.3 per cent

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[a] Rounds A and B were trial batches which differed in air content and slump from design tolerances; therefore, strength levels for these rounds are not necessarily comparable with those for Rounds C, D and E where air and slump were held to design limits indicated above.

crete slab, which is in an uncertain and variable condition of saturation. Of interest also is the fact that, while different aggregates may appear in standard tests to produce widely differing flexural strengths, those distinctions may disappear upon changes in moisture condition of the concrete.

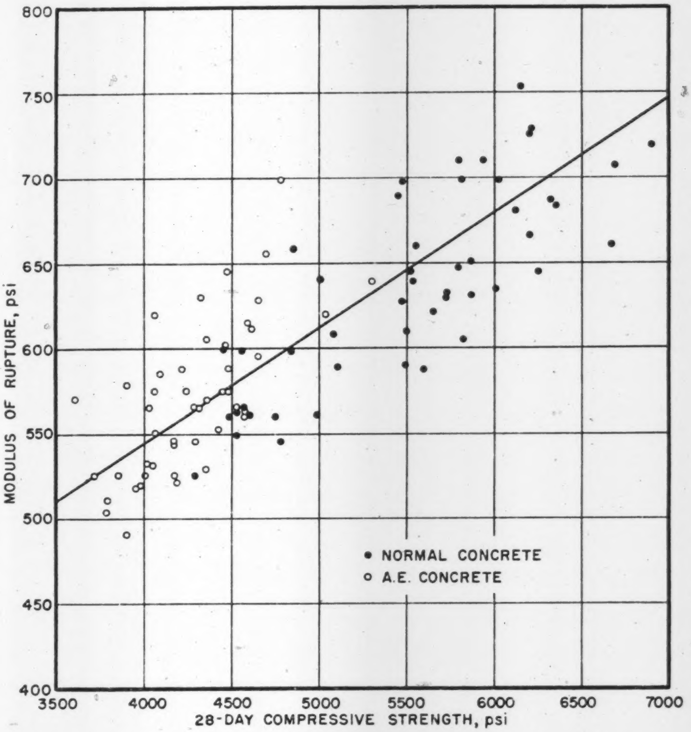

FIG. 7.—Relationship Between Flexural and Compressive Strengths for All Coarse Aggregates (Series 156).

## UNIFORMITY OF DUPLICATE TESTS ON SAME SPECIMEN

In one group of tests, where strength-producing properties of different cements were under study (1), duplicate flexural tests on the same beams were made to check within-batch uniformity. One 6 by 6 by 36-in. beam was molded from each of 60 batches of concrete, the beams being sufficiently long to provide two standard third-point loading tests on an 18-in. span. The beams were tested after 28 days of standard curing. Extreme care was taken to prevent the surfaces from drying between the first and second tests. The data are summarized in Table IV.

It is apparent that, even for the same specimen, where opportunities for differences in sample, molding, and curing were at an absolute minimum, the flexural test was subject to appreciable variation. The within-specimen coefficient of variation amounted to 5.3 per cent, with differences between two tests on the same specimen in some cases approaching 100 psi. It must be concluded that this variation depended upon the chance distribution of coarse aggregate or upon other lack of complete uniformity within the specimen. That differences were not due to changes in moisture condition between tests or to damage of the specimen in the first test is evidenced by the fact that over-all averages for the first and second breaks were essentially the same.

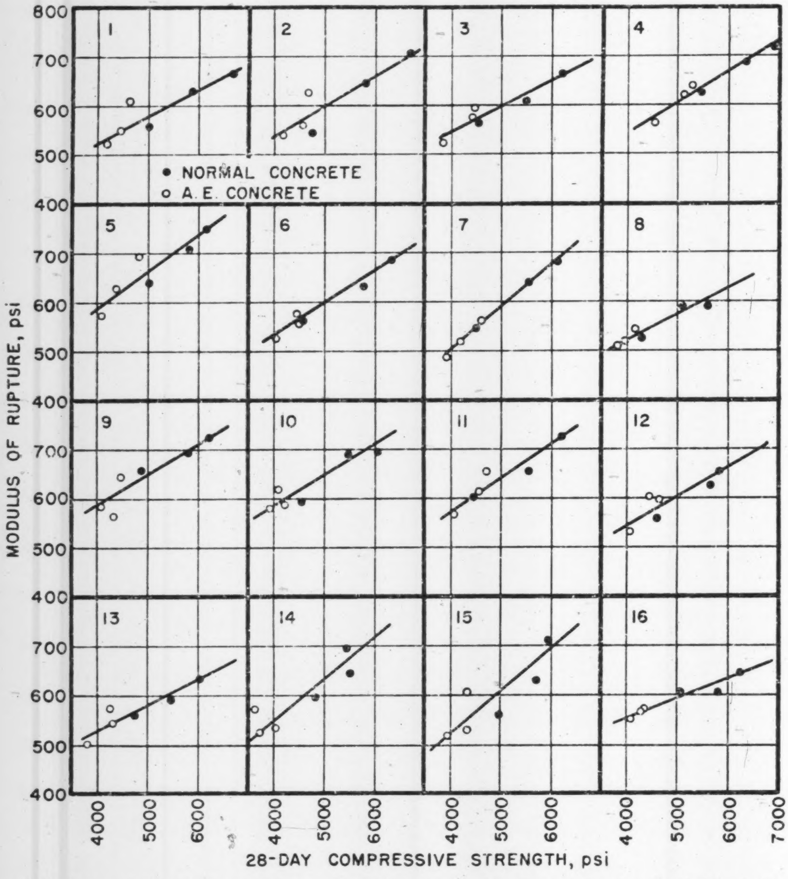

FIG. 8.—Relationships Between Flexural and Compressive Strengths for Individual Coarse Aggregates (Series 156).

For the 24 groups of 3 single specimens from batches C, D, and E (for which design criteria were maintained within required tolerances), the average batch-to-batch range in modulus of rupture was 56 psi. On the basis of the "range" formula for approximating the standard deviation (14) (the method used for analyzing within-specimen differences), the batch-to-batch standard deviation for single tests was 0.5907 × 56 or 33 psi. This is almost identical to the within-specimen standard deviation of 31 psi. Thus, under the careful laboratory control employed in these tests, variability of flexural strength was no greater between batches than between tests on the same specimen. Under conditions of field testing, which would be subject to many more sources of variation than duplicate tests on the same specimen in the laboratory, it appears that flexural strength differences would more often reflect inherent variability of the testing than actual differences in concrete quality.

### FLEXURAL *Versus* COMPRESSIVE STRENGTH

It has been shown that, over a wide range of different ingredients and proportions, only a very general relationship exists between compressive and flexural strengths (1,8). While flexural strength tends to increase as compressive strength increases, there is no consistent ratio relating the two.

Data from an earlier investigation (1), shown in Fig. 7, confirm the lack of a precise relationship between compression and flexure tests. That study involved concretes of 3 cement factors, with and without entrained air, made with each of 16 different coarse aggregates.

When, as shown in Fig. 8, data for the several coarse aggregates are separated, a good correlation between flexure and compression becomes evident. In some cases, the curves are relatively steep and, in others, quite flat. For example, in the case of aggregate No. 7, an increase of 3000 psi in compressive strength was accompanied by an increase of about 270 psi in modulus of rupture. For aggregate No. 16, on the other hand, the increase of 3000 psi in compression was accompanied by a change of only about 110 psi in flexure. Nevertheless, in virtually all cases it appears that, once a curve has been established for a given set of materials, the compressive strength test can be relied upon to provide a good indication of potential flexural strength. By establishing in advance what compressive strength is needed to assure the desired level of flexural strength, the compression test could be used for field control, thus avoiding use of the cumbersome and sensitive tests of beams.

### SUMMARY OF INDICATIONS OF TESTS

The data secured in these investigations provide indications, largely consistent with the work of other researchers, which may be summarized as follows:

1. For the concrete of moderate richness and slump used, measured modulus of rupture was unaffected by the position of the beam during loading in relation to its position as molded.
2. Failure to provide unrestrained bearing during loading did not significantly change the measured modulus of rupture, possibly because the specimens and bearing surfaces were sufficiently true that significant torsional stresses were not developed.
3. Introduction of nearly maximum torsion during loading significantly reduced the measured modulus of rupture.
4. Uneven bearing at the reaction and load points (but with the opportunity for torsion eliminated) did not significantly affect the measured modulus of rupture.
5. The third-point loading device pictured in ASTM Method C 78[4] gave lower strengths than the other acceptable device which employed rollers rather than rockers at the lower bearing points. This could have resulted from the induction of tensile stresses in the lower fibers due to a horizontal frictional component arising from deviation of the rockers from a vertical position.
6. Measured modulus of rupture decreased as depth of beam increased.
7. For concrete made with a hard quartz gravel, measured modulus of rupture was considerably lower for sawed than for molded beams.
8. Differences in modulus of rupture between duplicate tests on the same beam were appreciable, as evidenced by a within-specimen coefficient of variation of 5.3 per cent. Where all operations were under strict laboratory control, inherent variations in testing contributed as much (or more) to lack of uniformity in meas-

ured flexural strength as batch-to-batch differences in the concrete.

9. There was evidence that, other factors being comparable, thoroughly dry concrete produced higher flexural strength than moist concrete.

10. Partial drying of wet concrete caused substantial reductions in measured modulus of rupture, probably because of differential volume change which induced tensile stresses in the surfaces of specimens.

11. Large differences in flexural strength-producing properties of two coarse aggregates, shown for conditions of standard curing, tended to disappear when the concrete was permitted to dry. The strength differences remained negligible after the concrete was resoaked.

12. For each of 16 different coarse aggregates, there was an excellent relationship between modulus of rupture and compressive strength, although the flexural strength corresponding to a given compressive strength varied considerably among the different aggregates.

## CONCLUSION

This investigation corroborates other researches which have shown the measurement of flexural strength to be highly sensitive to variations in treatment and testing of specimens. The test for modulus of rupture (like that for compressive strength) is not a quantitative measure of load-carrying capacity of concrete in the field. Neither is it a desirable acceptance test for determining conformance with strength requirements for pavement construction.

The data indicate that, where flexural strength-producing properties are of major concern, entirely satisfactory control can be secured by establishing the relationship between flexural and compressive strengths from laboratory tests with the materials to be used, and thereafter using compression tests as the basis for acceptance.

### *Acknowledgments:*

These tests were conducted by James F. Shook, former Associate Research Engineer, R. D. Gaynor, Associate Research Engineer, and Herman L. Knoppel, Jr., Assistant Laboratory Supervisor, under the general direction of the authors. J. B. Blackburn, Associate Professor of Civil Engineering at the University of Maryland, assisted in the compilation of data and performed statistical analyses.

## REFERENCES

(1) Stanton Walker and Delmar L. Bloem, "Studies of Flexural Strength of Concrete—Part 1: Effects of Different Gravels and Cements," Joint Research Laboratory Publication No. 3, Nat. Sand and Gravel Assn. and Nat. Ready Mixed Concrete Assn.

(2) Stanton Walker and Delmar L. Bloem, "Studies of Flexural Strength of Concrete—Part 2: Effects of Curing and Moisture Distribution," *Proceedings*, Highway Research Board, Vol. 36 (1957); reprinted as Nat. Sand and Gravel Assn. Circular 69 and Nat. Ready Mixed Concrete Assn. Publication No. 72.

(3) P. E. Irick and J. B. Blackburn, "Application of Statistical Methods to Laboratory Concrete Freeze-Thaw Test Data," *Proceedings*, Highway Research Board, Vol. 34, p. 329 (1955).

(4) John Tucker, Jr., "Statistical Theory of the Effect of Dimensions and of Method of Loading Upon the Modulus of Rupture of Beams," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1072 (1941).

(5) C. P. Lindner and J. C. Sprague, "Effect of Depth of Beam Upon the Modulus of Rupture of Plain Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 1062 (1955).

(6) F. V. Reagel and T. F. Willis, "The Effect of the Dimensions of Test Specimens on the Flexural Strength of Concrete," *Public Roads*, Vol. 12, No. 2, Apr., 1931.

(7) W. F. Kellerman, "Effect of Size of Specimen, Size of Aggregate, and Method of Loading Upon the Uniformity of Flexural

Strength Tests," *Public Roads*, Vol. 13, No. 11, Jan., 1933.

(8) Clyde E. Kesler, "Statistical Relation between Cylinder, Modified Cube, and Beam Strength of Plain Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 1178 (1954).

(9) William C. Otto, "A Statistical Study on Flexural Strength of Concrete Beams," *Proceedings*, Am. Soc. Testing Mats; Vol. 57 (1957)

(10) Delmar L. Bloem, "Flexural Strength Tests of Sawed Concrete Beams," enclosed with Natl. Ready Mixed Concrete Assn. Technical Information Letter No. 106, July 11, 1956.

**(11)** D. O. Woolf, "Effect of Temperature and Moisture Content on Flexural Strength of Portland Cement Mortar," *Public Roads*, Vol. 15, No. 10, p. 248 (1934).

**(12)** W. F. Kellerman, "The Effect of Curing Conditions on the Strength of Concrete Test Specimens Containing Burnt Clay Aggregates," *Public Roads*, Vol. 188, No. 3, p. 53 (1937).

**(13)** W. F. Kellerman, "Tests of Concrete Containing Waylite," *Public Roads*, Vol. 24, No. 9, p. 244 (1946).

**(14)** "Manual on Quality Control of Materials," Am. Soc. Testing Mats. (Issued as separate publication *ASTM STP No. 15-C*).

# DISCUSSION

Mr. George Werner.[1] (*presented in written form*).—The authors' paper is a valuable addition to the published information on the effect of variations in testing procedures on the results of flexural strengths of concrete beams when tested by third-point loading. Testing engineers have known of the effects of various moisture conditions on the flexural strength of test beams since the early 1930's and have realized that meticulous care was necessary in molding, curing, and testing of the beam in flexure. However, the authors have called attention to the possible need of a flexural testing device that might be superior to that pictured in ASTM Method of Test C 78.[2] As I read in Method C 78 the use of this pictured device is not mandatory.

The authors state in the synopsis of their paper that the data indicate the flexural strength test is not suitable as a quantitative basis for acceptance or rejection. This is somewhat surprising when one considers some of their data that were obtained from tests made in a laboratory where control of molding, curing, and testing was excellent.

Table I shows that concordance of results for beams tested on a standard device, although not that of Method C 78, with the side, top or bottom of the beam in tension, was 3.9 and 3.7, 4.4 and 3.8, and 3.0 and 5.2 per cent respectively, when expressed as coefficient of variation, which I consider to be very good. Possibly the authors mean that the test for flexural strength should not be used for acceptance or rejection when conditions of proper curing and testing on an acceptable device have not been met.

With no desire to take issue with the authors' statement, I believe that the flexural strength test should be considered as a valuable tool and guide for the design or determination of the mix proportions for paving concrete and for determining when a road may be opened to traffic. The value of 550 or 650 psi has been used for many years as a means of design in the laboratory for paving mixes. On the construction job low flexural strengths have been a warning to the engineer that he has heeded by checking cement content, water content, air content or low curing temperatures of the concrete. The test also serves to indicate changes in the quality of the coarse aggregate. With proper compliance in the field with ASTM Method C 31[3] and by use of a suitable field beam tester of which there are at least three on the market at the present time, it is more convenient to make flexural strength tests than to make compressive strength tests.

The Bureau of Public Roads tested sawed beams, 8 in. wide by 10 in. in depth for the U. S. Engineers Office in 1944. These beams had been sawed by a

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[1] Highway Physical Research Engineer, U. S. Bureau of Public Roads, Washington, D. C.

[2] Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 – 49), 1955 Book of ASTM Standards, Part 3, p. 1323.

[3] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 – 55), 1955 Book of ASTM Standards, Part 3, p. 1314.

36-in. abrasive wheel. They were totally immersed in water 5 to 10 days before testing and some were tested with a sawed side in tension and the others were tested with a mortar surface in tension. Strengths were lower for the beams tested with a sawed surface in tension. Every testing engineer should recognize the possible damage to test beams by sawing. It seems reasonable that such damage and insufficient saturation before testing would cause less decrease in flexural strength of sawed beams with a cross section of 8 by 10 in. than in the results of tests of sawed beams of 3 by 4-in. cross-section as reported by the authors.

The highway engineer still regards the flexural strength test as an aid toward obtaining better concrete. The usual penalty when the designed flexural strength is not obtained on the job is a sleepless night for the inspector and a closer attention to control by the contractor.

MR. ALBERT T. GOLDBECK.[4]—About 1924 I had the very great privilege of occupying a desk next to Mr. H. M. Westergaard in the Bureau of Public Roads. He had been commissioned at that time to work on the development of a theory for the design of concrete pavements. I well recall he sat there many a day and said, "I wish I knew more mathematics," although as you probably know, he was one of the finest mathematicians of that time.

He finally developed a remarkable theory which has persisted up to the present with very little change. It is the basis for most of the theory that is now being used for concrete pavement design. There have been some modifications but modifications due primarily to the deforming effect of heavy traffic on the subbase. That effect has rather killed the idea that you can have a uniformly elastic subbase unless you provide an adequate and properly graded subbase material.

The point is that in Mr. Westergaard's theory use was made of modulus rupture as a measure of concrete strength. The maximum strength of the concrete was determined by means of a modulus of rupture test. So I would dislike to see the modulus rupture test discarded. I do not think that would be a step in the right direction.

Now I have not had an opportunity to study the results the author has described, so I cannot discuss those results in detail. But when the author was describing the relationship between modulus of rupture values as determined by the device they used as compared with others, a device presumably equal to that shown in the ASTM standard, I wondered if these different devices would necessarily give the same results. I do not know anything about their machine, but as you know, when you make a load test on a beam, you produce tension in the lower fibers, compression in the upper fibers. That tension in the lower fibers results in the outward rocking of the supports. Now suppose there is resistance to that outward rocking, due to high rolling resistance of the roller supports. If that is the case there will be a horizontal component acting toward the center which will tend to reduce the tension in the beam and the result will be an increase in the calculated modulus of rupture. That occurs to me as being a possible explanation of the difference in the modulus rupture values obtained by the Nat. Sand and Gravel Assn. method and those obtained with the apparatus depicted in the ASTM Standard.

Years ago there was a committee, I think Mr. F. V. Reagel of Missouri was chairman of it, commissioned to determine what was the proper kind of a laboratory test to make on beams. It used a device which had hangers at the ends to support the specimen. There could not possibly have been any horizontal component developed at the supports, the force at the reaction points must have been vertical. But this form of device was difficult to use.

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[4] Engineering Consultant, National Crushed Stone Assn., Washington, D. C.

Finally, from the standpoint of ease of making the test, the ASTM machine was devised and it gave essentially the same results as were obtained by the hanger method of suspension. So I am wondering all the more whether the difference in results obtained was not due to the rolling friction of the roller supports at the ends of the beams in the device used in the author's laboratory.

MESSRS. STANTON WALKER AND DELMAR L. BLOEM (*authors' closure*).—Mr. Werner and Mr. Goldbeck apparently derived the impression that this paper advocated the abandonment of flexural strength as a basis for concrete pavement design. Such was not the intention. Accepted design theories depend upon a knowledge or assumption of flexural strength for determining load-carrying capacity or selecting required thickness. Tests for modulus of rupture are necessary in establishing concrete proportions required to secure the desired strength level and in comparing strength-producing properties of different combinations of concrete ingredients. However, this work should be confined to the laboratory where testing conditions can be meticulously controlled. The test for modulus of rupture is too sensitive to extraneous factors, particularly moisture condition of specimens, to permit its use as a basis for the acceptance or rejection of concrete in the field.

The good reproducibility of test results for any given condition, mentioned by Mr. Werner, is hardly a basis for assuming that the test is dependable under usual field conditions. It means merely that conditions in the research were highly uniform for replications of a given variable. The significant feature is the variation from condition to condition, particularly with respect to moisture, which represents the important effect of slight differences in field testing conditions. One can well wonder whether the sleepless nights of inspectors, to which Mr. Werner refers, do not result more often from lack of reproducibility of field testing conditions than from actual deficiencies in concrete quality.

Mr. Werner's point about the effect of specimen size on the relationship between sawed and molded beams is well taken. It seems quite likely that the small size of the beams magnified the disadvantage of the sawed specimens. The data should be interpreted as illustrating a potentially important effect on measured strength rather than as giving a quantitative measure of strength differences.

Mr. Goldbeck offers a different hypothesis than the authors' to account for differences between the ASTM Method C78 loading device and the other standard apparatus. His suggestion that frictional forces created by the rollers could have produced compressive stresses which increased load-carrying capacity is inconsistent with data for the other test conditions. If frictional restraint could account for higher measured strength, then loading through fixed bearing plates should have increased the modulus of rupture instead of producing the same value as the apparatus with rollers. In any event, the principal criticism of the Method C78 device is its lack of stability, not its reduction of measured strength.

# WEAR TESTS ON CONCRETE USING THE GERMAN STANDARD METHOD OF TEST AND MACHINE*

By James L. Sawyer[1]

## Synopsis

In practice, a floor surface must withstand large wheel pressures brought about by heavy loads of small-wheeled carts being rolled across the floor.

It has been realized for many years that a test was needed to measure the resistance of concrete surfaces to wear. Much work has been done in this country by members of Subcommittee III-m of ASTM C-9 on Concrete and Concrete Aggregates and by others to develop a test method yielding reproducible results. The Germans, also, have been working on the same problem and have recently developed a machine and method for abrasion test which was adopted as Standard DIN 51951.

The German test duplicates a practical wear condition by rolling steel balls under pressure over the concrete surface. This results in the concrete surface being subjected to abrasion caused mainly by rolling and sliding friction accompanied by some pounding or beating force.

This paper reports work with the German standard machine using a slightly modified test method.

The test is sensitive to differences in mix and curing procedures. The results are reproducible and easily obtained. Results indicate that adequate curing is essential for good wear resistance. It would appear that some relationship exists between compressive strength and wear resistance.

The industrial demands that are placed on concrete surfaces make it imperative that these concrete surfaces be capable of withstanding many abrasive forces with a minimum of wear. In practice, a concrete floor surface must withstand compressive forces brought about by wheel pressures from heavily loaded carts and other equipment. As these small-wheeled carts are drawn across the floor, the floor is subjected to the moving pressure of the rolling wheels. At the same time, some sliding friction occurs as well as a beating or pounding force when surfaces are rough. This combination of destructive forces can result in an ever deepening and penetrating wear of the concrete surface.

It has been realized for many years that the resistance to wear of concrete was of great interest (**1**,**2**)[2] and that a reliable test was needed that was capable of measuring this resistance to wear. Much work has been done in this country by members of Subcommittee III-m of ASTM Committee C-9 on Concrete and Concrete Aggregates and by others to develop a test method yielding reproducible results. D. A. Abrams, in his paper on "Strength and Wear of Concrete" presented at the fourteenth annual convention of the American Concrete Inst. in 1918 reported work done with the Talbot-Jones Rattler (2).

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Research Chemist, Lone Star Cement Research Laboratory, Hudson, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1153.

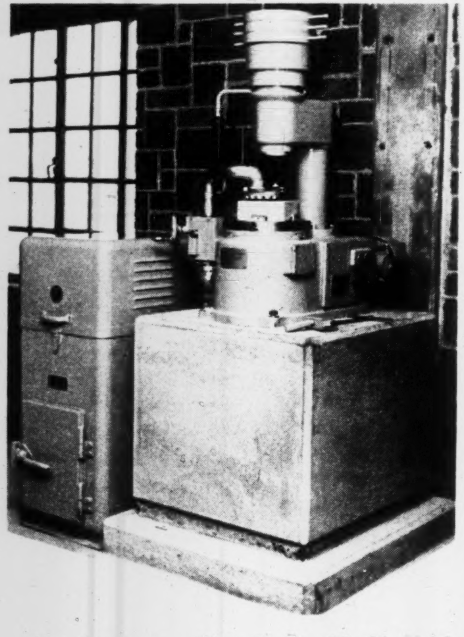


FIG. 1.—German Wear Testing Machine with Test Specimen in Place.

Various other methods have been used, such as sand blasting, grinding wheel, dressing wheel, Hughes bit, et cetera. It is felt that any test method that is considered should meet the following requirements:

1. The test specimen should be subjected to forces that are similar to those experienced in practice.
2. The test must be severe enough to cause deterioration of any concrete surface.
3. The test must be sensitive to variations in surface conditions.
4. The test specimens should be easy to make, store, and handle.
5. The test method should be easy to follow, and the cost and time of testing should not be excessive.
6. The test results must be reproducible.

In Germany wear tests have been going on, with work begun in 1936 to set up standards for hard concrete surfaces (3). This led in 1941 to the publication of a Standard Specification DIN 1100. Work was interrupted during the war, but beginning in 1950 the reorganized German Concrete Society continued the further development of the standard. The committee for measuring and testing of hard concrete was reactivated and various methods were considered.

Fritz Ebener developed a testing apparatus which subjected a concrete specimen to abrasion by rotating balls under pressure. Comparative tests were made on four of these machines in four laboratories in June of 1953. The results of these tests indicated that the test machine and test method were acceptable as a standard.

The German test duplicates a practical wear condition by rolling steel balls under pressure over the concrete surface. This results in the concrete surface being subjected to abrasion caused mainly by rolling pressure and sliding friction accompanied by some pounding or beating force.

The work presented in this paper was done with the German machine and a slightly modified test method.

### *Testing Machine:*

The Ebener testing machine corresponds to the requirements of Standard DIN 51951. As shown in Fig. 1, it consists essentially of a horizontal turn-

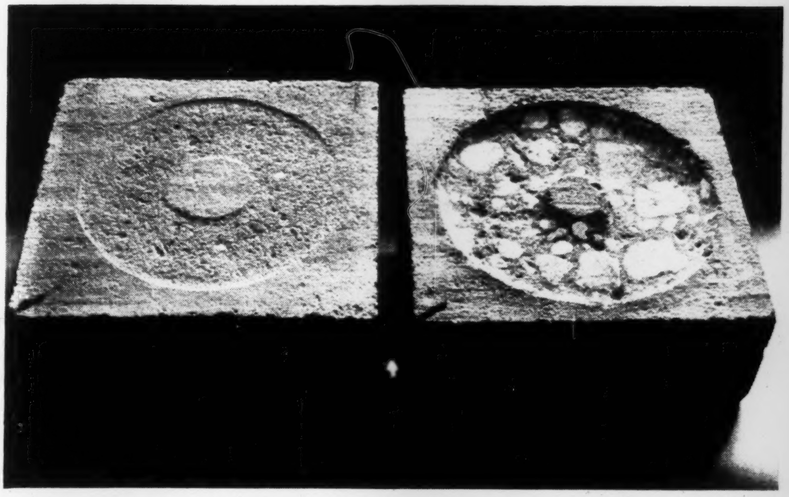
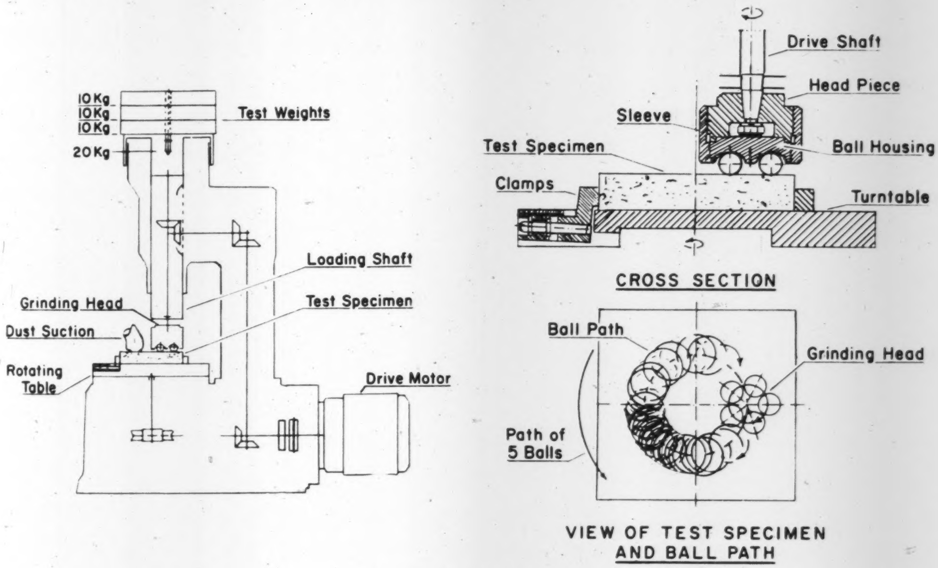


FIG. 2.—Worn Test Specimens After One Test Period and Ten Test Periods.


10 Kg
10 Kg
10 Kg
20 Kg
Test Weights
Loading Shaft
Grinding Head
Test Specimen
Dust Suction
Rotating Table
Drive Motor
Drive Shaft
Head Piece
Sleeve
Test Specimen
Ball Housing
Clamps
Turntable
Ball Path
Grinding Head
Path of 5 Balls

CROSS SECTION

VIEW OF TEST SPECIMEN AND BALL PATH

FIG. 3.—Wear Test Apparatus.

table on which the test specimen is placed and a loading press installed vertically in the head of the machine. A spherical head is attached to the loading press in such a way that the head can be rotated. The centers of the turntable and the spherical head are displaced in relation to each other by 40 mm. The turntable and the spherical head are contra-rotating, the relation of their speeds being 1:35. The spherical head holds five $\frac{23}{32}$-in. ball bearings running in a hardened raceway 33 mm in diameter. Due to the eccentricity of the spherical head and of the turntable, the balls rolling and sliding on the surface of the specimen subject a circular annular area of the concrete surface to wear (Figs. 2 and 3).

The weight of the applied load can be varied from 20 to 50 kg in increments of 10 kg. A specimen 6 by 6 in. in section and up to $2\frac{3}{4}$ in. in thickness can be used. This specimen is clamped to the turntable by two movable jaws located opposite to two fixed stops. Thus, by marking one corner of the specimen, it can be returned to its initial position after being removed from the machine upon completion of the various test periods. The dust abraded during the test is sucked off by a suction nozzle into a vacuum cleaner. The machine is equipped with an automatic device which stops the machine after 40 rotations of the base table (1400 rotations of the ball holder, approximately $1\frac{3}{4}$ min test time).

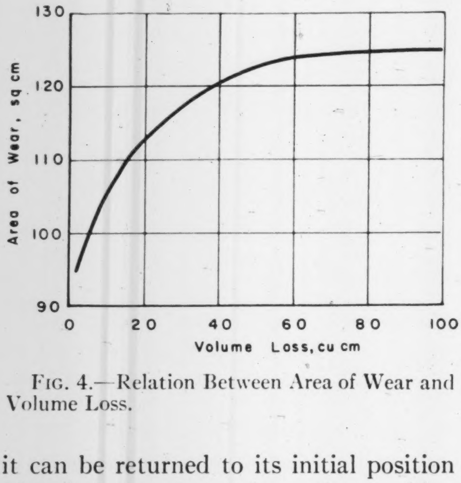


FIG. 4.—Relation Between Area of Wear and Volume Loss.

### *Testing Procedure:*

The testing procedure provides for ten test periods of 40 rotations of the table per period. The specimen is weighed before and after each test period and the loss in weight noted. Also the outside and inside diameters of the worn annular area are measured after each test period. From these measurements the abrasion area can be determined. With these data and the density of the test specimen, the volume of the abraded material and the average depth of wear can be calculated. The wear area is not constant, however it increases only as the balls penetrate deeper into the specimen. The relationship between volume abraded and area of wear is shown in Fig. 4. This relationship will apply to any specimen as long as the diameter of the raceway does not increase due to wear. From this curve the wear area for any given volume loss as shown in these tests can be found without the necessity of measuring the area. The wear area varies from a minimum of 95 sq cm to a maximum of 125 sq cm, representing a range of volume loss from 2 cu cm to 100 cu cm. The 100 cu cm volume loss represents an 8 mm depth of wear, which is about the maximum depth that can be obtained with this machine and is the limit of clearance between the spherical head and specimen. Any wear greater than 8 mm depth must be interpolated.

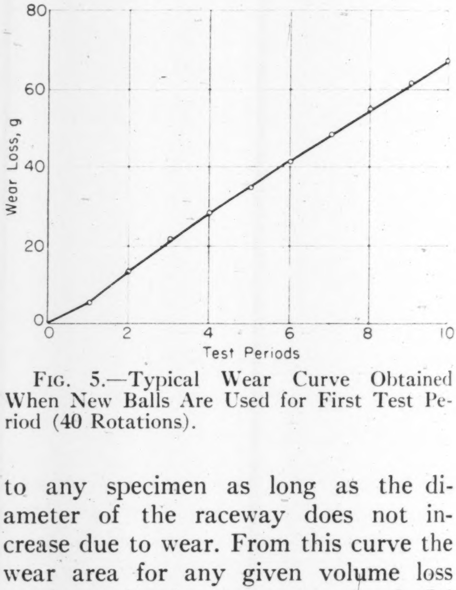


FIG. 5.—Typical Wear Curve Obtained When New Balls Are Used for First Test Period (40 Rotations).

The testing procedure calls for new balls at the beginning of each test. If this is interpreted to mean new balls for each test period, a total of 50 balls is required for each specimen tested. However, if new balls are used only at the beginning of tests on each specimen, then the first one or two test periods will show less wear than succeeding periods due to the changing condition of the balls. This results in a wear curve as shown in Fig. 5 in which the wear for the first one or two test periods is less than for the succeeding periods. This is the type of wear curve that was reported by E. Plassmann in *Zement-Kalk-Gips* (**3**), so it seems probable that the German testing was done with a new set of balls for each specimen, the balls being in a new and polished condition for the first test period and in a roughened condition for the remainder of the ten test periods. This could lead to an unjustified conclusion that the original surface of the test specimen was more resistant to wear than the concrete just under the surface. The author is of the opinion that before any valid conclusions can be drawn, the test must be conducted with the balls in a similar condition throughout the test.

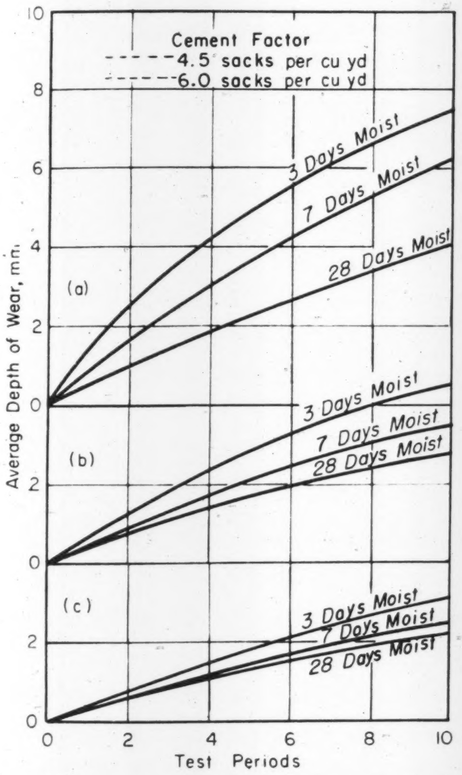


FIG. 6.—Effect of Curing Time on Wear Characteristics (2-in. Slump Concrete).

(*a*) 4.5 Sacks per cu yd. Water-cement ratio, 7.38 gal per sack.)

(*b*) 6.0 sacks per cu yd. Water-cement ratio, 5.64 gal per sack.

(*c*) 7.5 sacks per cu yd. Water-Cement Ratio, 4.60 gal per sack.

Results presented in this paper were obtained with balls that had been roughened prior to the first test period. This was achieved by using a new set of balls for one test period on a discarded specimen. When balls in a roughened condition were used for each test period, it was found that the wear curve did not always indicate that the surface was more resistant to wear than the interior of the specimen. In fact, the reverse was more often true (**4**).

The applied load for all tests was 50 kg. One type of sand and gravel graded from $\frac{1}{4}$ to $1\frac{1}{2}$ in. except where noted, was used for all specimens. The author realizes that the aggregate used affects the wearing qualities of concrete (**5**). However, for these tests only the type of cement, the cement content of the concrete, water ratio and the curing were varied. No comparison of the types of cement are attempted in this paper, although, data obtained with type I, type II and type III cements are reported. These results can be considered representative, but space does not permit the inclusion of results from all tests with each type of cement.

The test specimens were molded in 6

by 6-. . by 2½-in. deep steel molds. The concrete was rodded, screeded, and wood floated while plastic. After the concrete had stiffened, the surface was steel troweled. Care was taken to avoid working excess water to the surface.

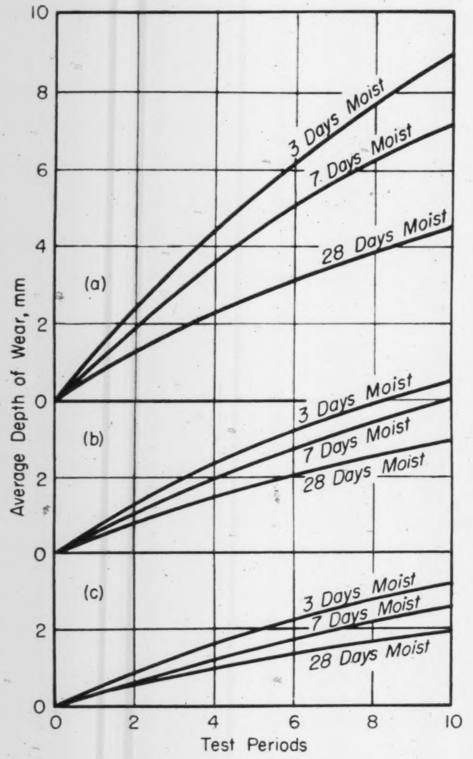


FIG. 7.—Effect of Curing Time on Wear Characteristics (6-in. Slump Concrete.

(*a*) 4.5 Sacks per cu yd. Water-cement ratio, 7.97 gal per sack.)

(*b*) 6.0 Sacks per cu yd. Water-cement ratio, 5.86 gal per sack.

(*c*) 7.5 sacks per cu yd. Water-cement ratio, 4.87 gal per sack.

The specimen was covered with a steel plate and damp burlap (supported above the surface of the specimen). After 24 hr in molds, the specimens were removed and stored in the fog room until the start of drying. Except where noted all specimens were dried at 100 F for seven days prior to testing. Density of specimens was determined by weighing in air and water immediately after removal from the fog room and subtracting weight loss after drying from the moist weight in air.

## RESULTS OF TESTS

The results of a typical series of tests are presented in Figs. 6 and 7. In each case the wear is represented by the average depth of wear in millimeters *versus* the number of test periods (40 rotations each). The wear could be expressed in other terms. Weight loss would be the simplest, requiring only the weighing of the specimen before and after each test. To convert to volume loss requires the assumption that the specimen had a uniform density throughout the full section. It is doubtful that this uniform density exists, as the temperature and time of drying is not sufficient to dry the interior completely. However, small differences in density do not produce significant errors in the final results. To convert from volume loss to average depth of wear requires only the area of wear. As already stated, this can be obtained from Fig. 3 more easily than by making precise measurements after each test.

### *Effect of Curing and Cement Content:*

Figure 6 presents the wear data obtained on 2-in. slump concretes using a type II cement. Three different cement contents were used (4.5, 6.0, and 7.5 sacks of cement per cubic yard of concrete). Specimens were cured moist for 3, 7, and 28 days prior to drying.

These wear curves clearly show that as cement content was increased wear was reduced. Also, significant reduction in wear occurred as days of moist curing was increased. It can be seen that adequate curing was important to all three mixes but became more important as the cement content was reduced.

Figure 7 represents the same concretes with a 6-in. slump. The increase in slump had little effect on the richer mixes but a marked reduction in wear resistance occurred in the mix containing the 4.5 sacks per cubic yard.

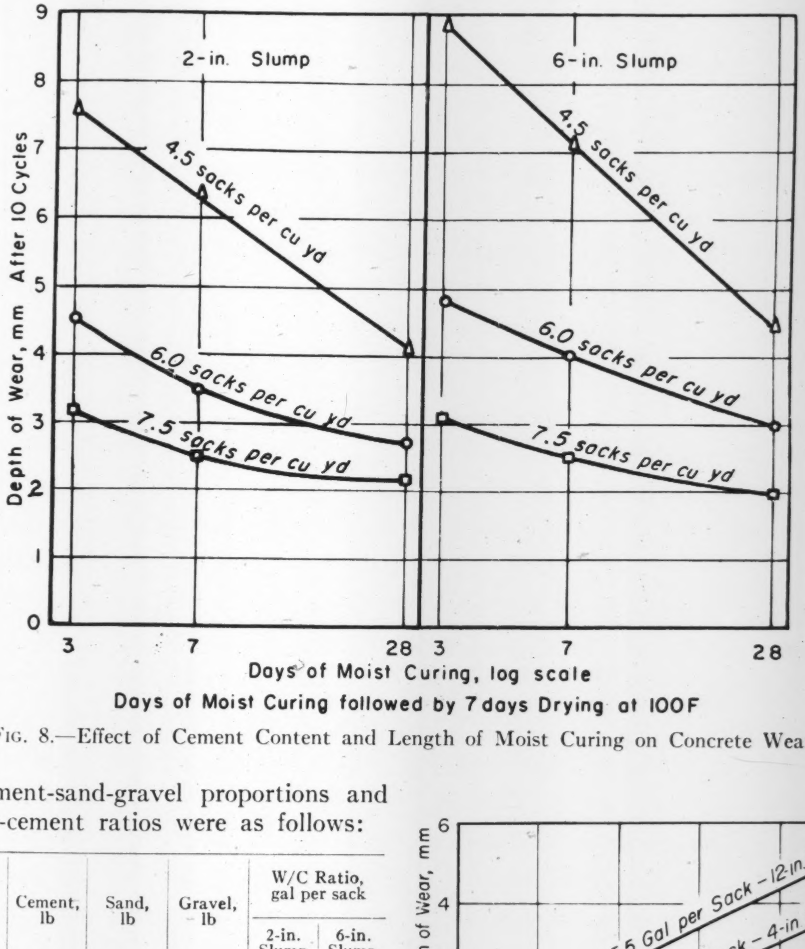


FIG. 8.—Effect of Cement Content and Length of Moist Curing on Concrete Wear.

Cement-sand-gravel proportions and water-cement ratios were as follows:

| Sacks per cu yd | Cement, lb | Sand, lb | Gravel, lb | W/C Ratio, gal per sack | |
|---|---|---|---|---|---|
| | | | | 2-in. Slump | 6-in. Slump |
| 4.5 | 37.5 | 102 | 186 | 7.38 | 7.97 |
| 6.0 | 50.0 | 88 | 192 | 5.64 | 5.86 |
| 7.5 | 62.5 | 74 | 196 | 4.60 | 4.87 |

In both the 2-in. and 6-in. slump concretes, the top surface of the concrete was more susceptible to wear than the concrete just under the surface.

A résumé of depths of wear after 10 test periods is given in Fig. 8.

*Effect of Water-Cement Ratio:*

Figure 9 shows more clearly the result of changing the water-cement ratio

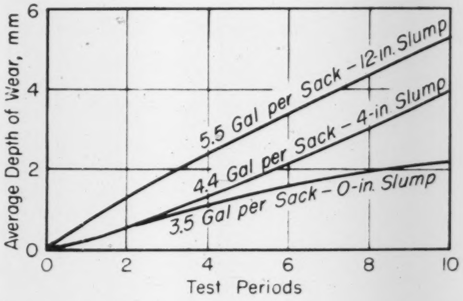


FIG. 9.—Effect of Changing Water-Cement Ratio on Wear, Type III Cement, Moist Cured 2 days, Dried 7 days.

These specimens were made with type III cement, sand, and $\frac{3}{8}$-in. gravel, proportioned to $1{:}1\frac{1}{2}{:}2$ by volume. Water-cement ratios of 5.5, 4.4, and 3.5 gal of water per sack of cement were used. The specimens were cured moist for two days and then dried for seven days at 100 F.

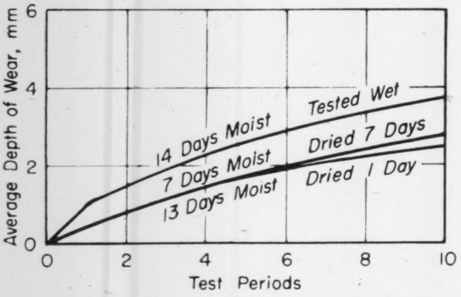


FIG. 10.—Effect of Drying on Wear-Type I Cement, Water-Cement Ratio = 4.5 gal per sack.

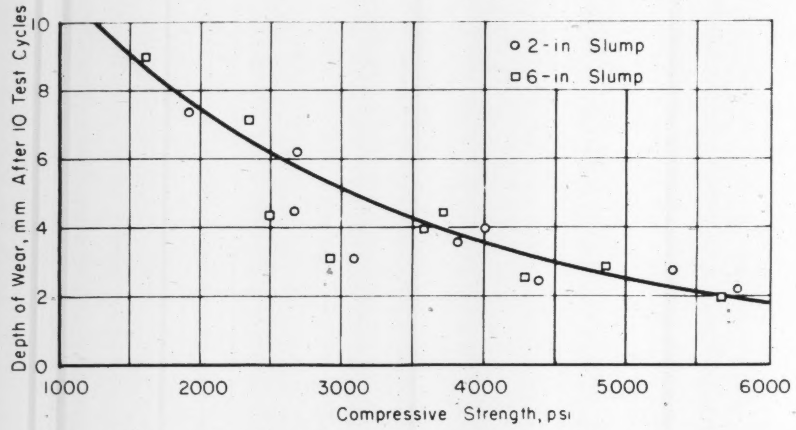


FIG. 11.—Relation Between Compressive Strength and Wear.

These results show that as the water-cement ratio was reduced the wear resistance increased. It is of interest to note that the top surface of the 3.5 gal per sack mix was no more resistant to wear than the surface of the 4.4 gal per sack mix. Once the top surface was passed, the increased wear resistance of the 3.5 gal per sack mix was obvious. It is possible that in troweling this stiff mix, a sufficient amount of water may have been brought to the surface to approximate the surface of the 4.4 gal per sack specimen. If we can assume that this was the case, then it clearly points out the danger of improper final troweling of a floor surface.

### *Effect of Drying:*

It was realized that the amount of moisture in the specimen at the time of test would be of some importance. Duplicate specimens were prepared using a mix $1{:}1\frac{1}{2}{:}2$ by volume of type I cement, sand, and $\frac{3}{8}$-in. gravel. All specimens were tested at the age of 14 days. One specimen was cured moist for the entire 14 days and was tested in a moist condition. A second specimen was dried at 100 F for one day after 13 days of moist curing. The third specimen was dried seven days at 100 F after seven days of moist curing. The damp specimen, even though it had received more moist curing than either of the other specimens, gave the least resistance to wear. Drying for one day gave a marked improvement in wear resistance. In fact, as can be seen from Fig. 10, this one day of drying gave less wear than the specimen that had been dried for seven days after seven days of moist curing. This improvement, which was probably due to the longer moist curing, was not noticeable at the surface of the slab. It would appear that drying of the surface is as important as additional moist curing.

TABLE I.—TYPICAL VARIATIONS IN WEAR TEST RESULTS OBTAINED ON NINE DUPLICATE TEST SPECIMENS (6 SACK CONCRETE).

| Test Specimen | Loss per Test Period, g | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Period 1 | Period 2 | Period 3 | Period 4 | Period 5 | Period 6 | Period 7 | Period 8 | Period 9 | Period 10 |
| No. 1 | 10.0 | 10.0 | 9.0 | 8.0 | 8.0 | 7.0 | 7.0 | 7.0 | 7.0 | 7.5 |
| No. 2 | 8.0 | 9.5 | 7.5 | 9.0 | 9.0 | 8.0 | 7.0 | 7.0 | 7.5 | 7.0 |
| No. 3 | 9.5 | 9.5 | 9.0 | 8.0 | 7.0 | 6.0 | 7.0 | 7.0 | 5.0 | 5.0 |
| No. 4 | 10.0 | 8.0 | 6.5 | 6.0 | 6.5 | 6.0 | 6.0 | 5.5 | 5.0 | 4.0 |
| No. 5 | 8.0 | 8.0 | 7.5 | 8.5 | 6.0 | 5.5 | 6.0 | 6.0 | 4.5 | 5.5 |
| No. 6 | 10.0 | 8.5 | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 | 7.0 | 7.0 | 7.5 |
| No. 7 | 10.0 | 8.5 | 7.0 | 6.0 | 6.0 | 5.0 | 5.0 | 5.0 | 5.0 | 5.5 |
| No. 8 | 13.0 | 10.5 | 9.0 | 8.0 | 7.5 | 7.0 | 6.5 | 7.0 | 6.0 | 7.0 |
| No. 9 | 13.5 | 11.0 | 8.0 | 7.0 | 6.0 | 6.0 | 6.0 | 6.0 | 6.0 | 6.0 |
| Avg. | 10.2 | 9.3 | 7.8 | 7.4 | 6.9 | 6.3 | 6.3 | 6.4 | 5.9 | 6.1 |
| Std. dev. | 1.8 | 1.0 | 0.9 | 1.0 | 1.0 | 0.8 | 0.6 | 0.7 | 1.0 | 1.1 |
| ACCUMULATIVE LOSS, G | | | | | | | | | | |
| No. 1 | 10.0 | 20.0 | 29.0 | 37.0 | 45.0 | 52.0 | 59.0 | 66.0 | 73.0 | 80.5 |
| No. 2 | 8.0 | 17.5 | 25.0 | 34.0 | 43.0 | 51.0 | 58.0 | 65.0 | 72.5 | 79.5 |
| No. 3 | 9.5 | 19.0 | 28.0 | 36.0 | 43.0 | 49.0 | 56.0 | 63.0 | 68.0 | 73.0 |
| No. 4 | 10.0 | 18.0 | 24.5 | 30.5 | 37.0 | 43.0 | 49.0 | 54.5 | 59.5 | 63.5 |
| No. 5 | 8.0 | 16.0 | 23.5 | 32.0 | 38.0 | 42.5 | 48.5 | 54.5 | 59.0 | 64.5 |
| No. 6 | 10.0 | 18.5 | 25.0 | 31.5 | 38.0 | 44.5 | 51.0 | 58.0 | 65.0 | 72.5 |
| No. 7 | 10.0 | 18.5 | 25.5 | 31.5 | 37.5 | 42.5 | 47.5 | 52.5 | 57.5 | 63.0 |
| No. 8 | 13.0 | 23.5 | 32.5 | 40.5 | 48.0 | 55.0 | 61.5 | 68.5 | 74.5 | 61.5 |
| No. 9 | 13.5 | 24.5 | 32.5 | 39.5 | 45.5 | 51.5 | 57.5 | 63.5 | 69.5 | 75.5 |
| Avg. | 10.2 | 19.5 | 27.3 | 34.7 | 41.7 | 47.9 | 54.2 | 60.6 | 66.5 | 72.6 |
| Std. dev. | 1.8 | 2.6 | 3.2 | 3.6 | 4.0 | 4.5 | 5.0 | 5.5 | 6.2 | 7.0 |
| Std. dev. per test period. | 1.8 | 1.3 | 1.1 | 0.9 | 0.8 | 0.8 | 0.7 | 0.7 | 0.7 | 0.7 |

These tests clearly indicate that any floor that is subject to heavy traffic while in a moist condition is more liable to show wear than the same floor maintained in a dry condition. This, of course, is well known, and the data in Fig. 10 therefore furnish an indication that this test method gives results that are comparable to wear observed in practice.

*Strength-Wear Relationship:*

From the same batches of concrete for the wear test specimens (Figs. 6 and 7), 6 by 12-in. cylinders were made. These cylinders were moist cured for 3, 7, and 28 days, then broken in compression. The relationship between these compressive strength results and the wear results is presented in Fig. 11. The relationship between wear and compressive strength is curvilinear. It must be noted, however, that all points do not fall on the curve. It would appear that compressive strength cannot be taken as a direct measure of wear, but rather as an indication.

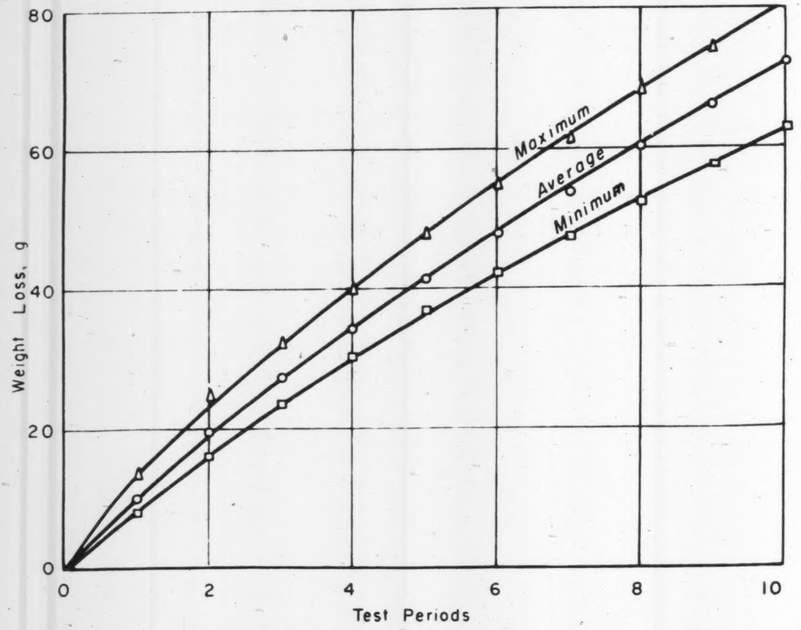


FIG. 12.—Typical Spread in Results—Maximum, Minimum and Average Weight Loss for Nine Duplicate Specimens.

*Reproducibility of Results:*

Three specimens were made from each of three duplicate batches of concrete, using 6.0 sack concrete in 2- and 6-in. slumps. The specimens were moist cured for 28 days and then dried at 100 F for seven days. Each group of nine duplicate specimens was made with a different cement, type I, type II and type III cements were represented. The wear was not the same for all cements, however, the variations of the duplicate specimens were within the limits normally experienced in other laboratory tests made on concrete. A representative series of results from these tests, with the wear reported as weight loss, are given in Table I. We can observe from these data that, while individual losses from one slab to the next during any single test period are somewhat variable, these variations are not cumulative but instead tend to cancel out if enough test cycles are run.

As can be seen from Fig. 12, the spread of wear from the least to the most wear for any one cycle is not excessive.

These tests were made at one laboratory only. The German tests made on four machines in four different laboratories, however, showed a similar spread in results (3).

## CONCLUSIONS

From the data presented it appears that the Ebener test machine subjects the test specimen to wear due to forces that are similar to those experienced by a floor surface in service. The test is capable of measuring wear on very resistant concrete surfaces and can give relative values for less wear-resistant surfaces. The test is sensitive to differences in surface conditions as well as conditions under the top surface. Thus it can ascertain the effect of varying finishing procedures, curing and drying conditions, mix proportions, and differences in aggregates and cements. The test specimens are small and easily prepared. The test can be easily and quickly run and the cost of testing is not excessive. The test gives results that are reproducible and easily interpreted.

Not enough data are as yet available for any definite conclusions to be drawn as to how much wear with this testing procedure would indicate a satisfactory or unsatisfactory floor under specified conditions of use.

However, the results indicate that, if the maximum wear resistance is desired from any concrete, the curing must be adequate. Adequate curing is important in all mixes, but it is of prime importance in the leaner mixes.

The increased wear due to surface moisture indicates that the expected moisture condition of the floor surface, when in service, must be considered in designing a suitable floor.

While it would appear that a definite relationship exists between compressive strength and wear resistance, the compressive strength of concrete alone cannot take into account the other factors that affect the wear resistance of a concrete surface.

## REFERENCES

(1) D. A. Abrams, "A Method of Making Wear Tests of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. XVI, Part II, p. 194 (1916).

(2) D. A. Abrams, "Effect of Time of Mixing on Strength and Wear of Concrete," *Proceedings*, Am. Concrete Inst., Vol. XIV, pp. 22–92 (1918).

(3) E. Plassmann, "Method of Testing Hard Concrete Surfaces Through the Pressure of Rolling Balls," *Zement-Kalk-Gips*, May 1954, pp. 213–216.

(4) Louis Schuman and John Tucker, Jr., "A Portable Apparatus for Determining the Relative Wear Resistance of Concrete Floors," *Journal of Research*, Nat. Bureau of Standards, Vol. 23, November, 1939, p. 560.

(5) F. L. Smith, "Effect of Aggregate Quality on the Resistance of Concrete to Abrasion," Papers on Cement and Concrete, Am. Soc. Testing Mats. (1956) (Issued as separate publication *ASTM STP No. 205.*)

# A STATISTICAL STUDY OF FLEXURAL STRENGTH OF CONCRETE BEAMS*

By William C. Otto[1]

*Purpose and Scope:*

The purpose and scope of the study of which this paper represents a part is to set reasonable limits for flexural strength of concrete pavement for military airfields. Reasonably high flexural strength of concrete for such fields is a necessity, mainly for the reason that inactivation of fields for repairs interrupts training schedules and the nonuse of very costly aircraft. This factor outweighs all others in importance. Extreme conditions of loading of the concrete slab by new types of military aircraft, high-pressure tires, and the cumulative effects of heat from jet engines require flexural strength higher and less variable than that required prior to the operations of jet planes. Good flexural strength helps to reduce the slab thickness without sacrifice of wheel load capacity. Restrained warping stresses are reduced as the slab thickness is diminished. The damaging effects of fatigue by repetitious, dynamic loading of the slab are also reduced as the slab thickness is decreased. The thinner the slab, the less steel and cement are required. These materials become critical at times and should be conserved. In Navy construction, the recent trend has been to reduce slab dimensions and to increase and render more uniform the flexural strength of concrete in airfield pavements.

The real engineering problem is to determine the proper balance from an economic standpoint of two separate factors that control the bearing capacity of concrete pavement. These two factors are (1) quality and flexural strength of concrete and (2) the thickness of slab and firmness of earth support. It never is real economy to rely on either factor (1) or (2) exclusively.

The variability of flexural strength of concrete for airfield pavement is considered in this paper. Another factor requiring study is the relation between 7-day and 28-day flexural strength tests of concrete beams wet cured. The specifying of good flexural strength is of paramount importance since an overloaded concrete pavement fails in flexure.

In this general study of flexural strengths of concrete beams, the relation between flexural and compressive strength will be included. Thus far, data pertaining to this topic are scant. The study also will include an evaluation and examination of the separate factors that control or influence flexural strength of concrete and its degree of variability. The combined influence of all variable factors that are traceable both to construction procedures and to testing techniques is involved.

*Source of Data:*

The data presented herein comprise

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* Presented at the Fifty-ninth Annual Meeting of the Society, June 17–22, 1956.

[1] Civil Engineer, U. S. Army Engineers District, Corps of Engineers, Detroit, Mich. Formerly Department of the Navy, Bureau of Yards and Docks, Washington, D. C.

the first increment of those of this general nature which are slowly being accumulated. Those considered in this paper were obtained from the records of the Sixth U. S. Naval District (Southeastern United States) and pertain to eight airfield projects, all located near the seacoast. All of the data relate to airfield pavements and none to highways, roads, or streets. The flexural strength tests were made in accordance with ASTM test procedures.[2] Most of the data were obtained by firms of consulting engineers, commercial testing laboratories, and by Navy civilian personnel.

Widely varying materials were used in the different projects. The period of construction covered is approximately four years under varying weather conditions. During this time, improvements in project specifications were made. There was considerable haste in construction during this period, owing to military requirements. Altogether there are over 1000 beam tests included in this progress report. The separate reports which furnish the data include average values for groups of three or more and of individual beams at both 7- and 28-day test periods. The number of tests per project for groups of beams varied from 19 to 64 groups while the number of individual beam tests per project varied from 45 to 241. The concrete pavement represented by the data includes runways, taxiways, parking aprons and warm-up aprons. Each project involved the construction of 50,000 sq yd or more of concrete pavement. The test data reflect the influence of variations of materials, climate, contractor's procedures, and testing techniques.

Table I gives a partial description of each of the eight projects included in this paper.

TABLE I.—DESCRIPTIONS OF PROJECTS.

| Project | Type of Construction | Required Los Angeles Wear of Coarse Aggregates, per cent | Design Mix (Yield 1 cu yd) | | | | |
|---|---|---|---|---|---|---|---|
| | | | Cement, lb | Coarse Aggregate, lb | Fine Aggregate, lb | Water, gal | Slump of Mix, in. |
| No. 1 | Parking Apron, LTA Mooring, and Taxiways | 40 | 611 | 1948 | 855 | 32.8 | .. |
| No. 2 | Warm-Up Apron | 45 gravel<br>40 stone | 587 | 2259 | 995 | 31.0 | 2 |
| No. 3 | Parking Apron | 45 | 564 | 2101 | 1086 | 33.0 | 1¼ |
| No. 4 | Runway and Taxiway | 50 | 533 | 2316 | 1074 | 30.0 | 1¼ |
| No. 5 | Runways and Apron | 45 | 611 | 1894 | 1066 | 34.13 | 2 to 3 |
| No. 6 | Runways, Taxiways, and Apron | 45 | 689 | 2207 | 1006 | 32.0 | 1 |
| No. 7 | Runway and Taxiway | 45 | 517 | 2278 | 1051 | 31.0 | 1¼ |
| No. 8 | Runways, Taxiways, and Aprons | 45 | 590 | 2319 | 931 | 32.0 | ¾ to 2½ |

### *Previous Statistical Studies Applied to Concrete Testing:*

The methods described in the ASTM Manual on Quality Control prepared by ASTM Committee E-11[3] were used as a general guide and pattern. This is a very helpful guide in the application of statistical methods of analysis to problems of construction. A later publication by Himsworth[4] utilized the same

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[2] Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 – 49), Part 3, p. 1051.

[3] ASTM Manual on Quality Control of Materials, Am. Soc. Testing Mats. (1951). (Issued as separate publication *ASTM STP No. 15-C*.)

[4] Francis Robert Himsworth, "The Variability of Concrete and its Effect on Mix Design," Paper No. 5950, *Proceedings*, Institution of Civil Engineers, Part 1 (General), Vol. 3. No. 2, Mar., 1954.

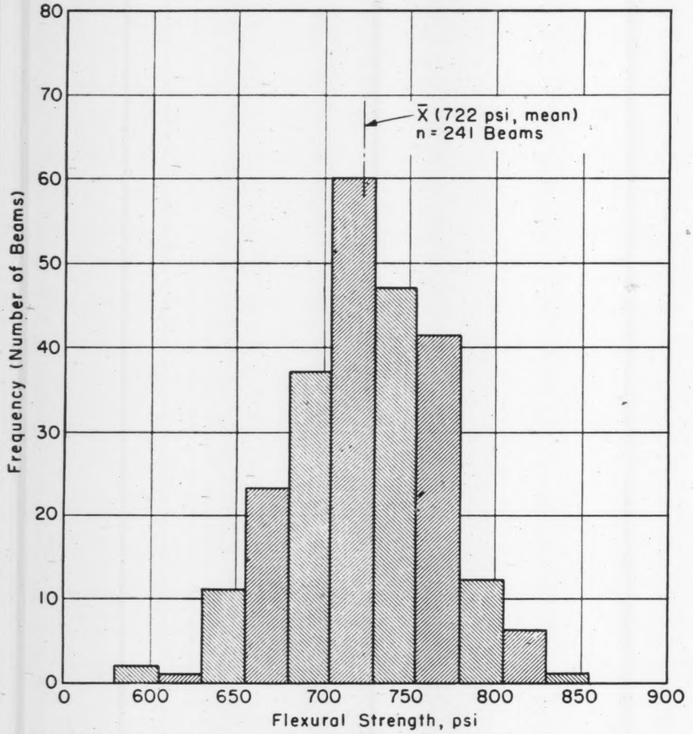

FIG. 1.—Typical Individual Strengths—28-Day Test Frequency Distribution Curve. Project No. 5.

methods of this committee. Further utilization was made by D. L. Bloem.[5] There are various other publications which have extended the application of statistical procedures or methods described by Committee E-11, but it seems that flexural strength data have received relatively little consideration.

*Project Descriptions, Definitions and Symbols:*

Descriptions of the projects are given in Table I.

The notations, formulas, and expressions used in this paper are the following:

$\bar{X}$ = The average (arithmetic mean), the sum of all of the observed values of a project, divided by their number $n$,

$n$ = The number of observed values (observations),

$\sigma$ = The standard deviation, the root-mean-square deviation of the observed values from their average,

$R$ = Range, the difference between the largest and the smallest observed values,

$X$ = A single observed value of a measurable quantity,

$k$ = Number of groups,

$\bar{\bar{X}}$ = Average of a set of group averages,

$\bar{L}$ = Average of the minimum values of groups,

$\sigma_L$ = Standard deviation of minimum values of groups, and

$v_L$ = Coefficient of variation of the minimum values of groups.

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[5] Delmar L. Bloem, "Studies of Uniformity of Compressive Strength Tests of Ready Mixed Concrete," ASTM BULLETIN, No. 206, May 1955, p. 65 (TP113).

Average (arithmetic mean)

$$\bar{x} = \frac{\Sigma X}{n} \quad \ldots\ldots\ldots\ldots \quad (1)$$

Standard Deviation

$$\sigma = \sqrt{\frac{\Sigma X^2}{n} - \left(\frac{\Sigma X}{n}\right)^2} \quad \ldots\ldots \quad (2)$$

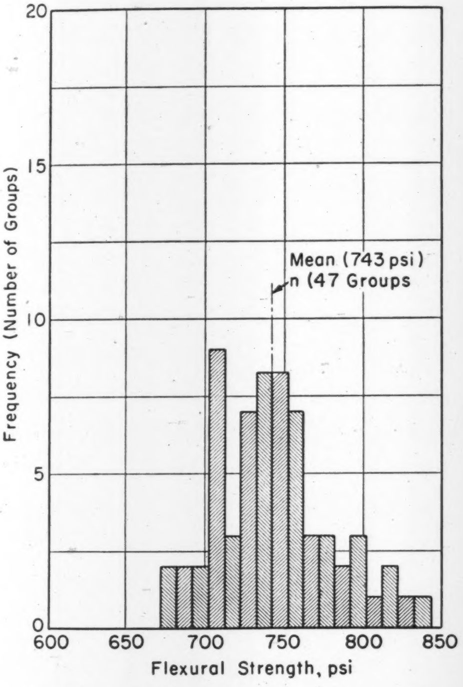

FIG. 2.—Typical Group Average Strengths—28-Day Tests Frequency Distribution Curve. Project No. 5.

Coefficient of Variation

$$v = 100 \frac{\text{Standard Deviation}}{\text{Arithmetic mean (Avg)}} \quad \ldots \quad (3)$$

$$\text{Control Limits} = \bar{\sigma}\left(1 \pm \frac{t}{\sqrt{2n}}\right) \quad \ldots \quad (4)$$

for standard deviation, where $\bar{\sigma}$ is an average of all standard deviations considered.

The expression,

$$R' = \overline{X} \pm t\sigma \quad \ldots\ldots\ldots\ldots \quad (5)$$

can be used to compute upper and lower tolerance limits, symmetrical about $X$, based on a total of $n$ observations.

Here, $t$ is a predetermined constant. When it is zero, $R'$ reduces to $X$. For controlled conditions Committee E-11 found no instance in data studied, and for which data $n$ exceeded 100 in all cases that at least 96 per cent of the observations did not fall within the limits obtained with $t$ equal to 3.

Theoretically, for a normal distribution 99.7 per cent of all observations fall within the interval that has for its limits the mean minus 3 standard deviations (lower limit) and the mean plus 3 standard deviations (upper limit). Hence, if the distribution for flexural strength of concrete beams follows the normal law, then Eq 5 becomes a yardstick for indicating the expected extent of variation.

## PRESENTATION OF DATA

### *Statistical Grouping of Data:*

The test data were condensed by grouping the observed flexural and compressive strengths into ordered classes. Each ordered class is a definite interval along a scale of measurement (flexural strength in psi) and is called a "cell." The data from each project was thus condensed and then plotted in graphical form. The height of each cell is equal to the number of observed values within the range of the cell. Figures 1 and 2 are typical frequency distributions for individual projects. The next step in the condensing and analysis of the flexural strength test data was the computation of the mean (average), the standard deviation, and the coefficient of variation for individual beam tests, group averages, and the minimum values of a group. Summaries are shown in Tables II, III, and IV.

The comparisons that can be made from the computed values of Tables II, III, and IV are as follows: Consider project No. 3—in Table II for individual beams and for $n = 192$ (project 3) $\bar{X}$ is 740 and $\sigma$ is 72 psi. This number, 192, is comprised of 64 groups, each having 3 beams and in Table III. for the 64 groups (project 3) $\bar{X}$ is 743 and $\sigma_{\bar{X}}$ is 37 psi. Table IV shows that for the 64 individual minimum flexural strengths within each of the 64 groups of 3 beams each, $\bar{L}$ is 713 and $\sigma_{\bar{L}}$ is 36 psi.

TABLE II.—FLEXURAL STRENGTHS—INDIVIDUAL BEAMS (28-DAY TESTS).

| Project | Individual Maximum, psi | Individual Minimum, psi | $n$ | $\bar{X}$, psi[a] | $\sigma$, psi[a] | $v$, per cent[a] |
|---|---|---|---|---|---|---|
| No. 1.... | 821 | 544 | 64 | 711 | 51 | 7.2 |
| No. 2.... | 816 | 512 | 45 | 681 | 64 | 9.4 |
| No. 3.... | 894 | 651 | 192 | 740 | 72 | 9.7 |
| No. 4.... | 866 | 672 | 57 | 770 | 47 | 6.1 |
| No. 5.... | 834 | 582 | 241 | 722 | 45 | 6.2 |
| No. 6.... | 925 | 625 | 115 | 748 | 59 | 7.9 |
| No. 7.... | 899 | 602 | 73 | 762 | 68 | 8.9 |
| No. 8.... | 824 | 470 | 238 | 679 | 82 | 12.0 |

TABLE III.—FLEXURAL STRENGTHS—ALL GROUP AVERAGES (28-DAY TESTS).

| Project | Maximum Group Average, psi | Minimum Group Average, psi | Number of Groups | Number of Observations in a Group | $\bar{X}$, psi[a] | $\sigma_{\bar{X}}$, psi[a] | $v_{\bar{X}}$, per cent[a] |
|---|---|---|---|---|---|---|---|
| | | | $k$ | $n$ | | | |
| No. 1.... | ...[b] | ... | ... | ... | ... | ... | ... |
| No. 2.... | ...[b] | ... | ... | ... | ... | ... | ... |
| No. 3.... | 836 | 675 | 64 | 3 | 743 | 37 | 5.0 |
| No. 4.... | 837 | 713 | 19 | 3 | 770 | 29 | 3.8 |
| No. 5.... | 769 | 657 | 47 | 6[c] | 723 | 28 | 3.8 |
| No. 6.... | ...[b] | ... | ... | ... | ... | ... | ... |
| No. 7.... | 821 | 690 | 18 | 4[d] | 765 | 35 | 4.6 |
| No. 8.... | ...[b] | ... | ... | ... | ... | ... | ... |

[a] Computed by grouping of observations into classes intervals.

[b] Beams were not tested in groups.

[c] Six values per group except, nine groups of 4 per group and two groups of 3 per group.

[d] Four values per group except, six groups of 3 per group and four groups of 5 per group.

In group testing, the standard deviation tends to be much less than that obtained from individual tests, as would normally be expected. It is the magnitude of this difference in the two $\sigma$ values thus obtained that is of special interest here. However (see Table IV), the $\sigma$ for all individual minimum values of the groups tends to compare well with that for the group averages (Table III).

*Analysis of Data:*

To determine whether the beam test data are indicative of controlled conditions, the observed percentages of observations in the range, $\bar{X} \pm t\sigma$ were computed for flexural strengths of individual beams.

TABLE IV.—FLEXURAL STRENGTHS—ALL MINIMUM BEAMS IN ALL GROUPS (28-DAY TESTS).

| Project | Greatest Individual Minimum, psi | Least Individual Minimum, psi | Number of Groups | Number of Observations | $\bar{L}$, psi[a] | $\sigma_{\bar{L}}$, psi[a] | $v_{\bar{L}}$, per cent[a] |
|---|---|---|---|---|---|---|---|
| | | | $k$ | $n$ | | | |
| No. 1.... | ... | ... | ... | ... | ... | ... | ... |
| No. 2.... | ... | ... | ... | ... | ... | ... | ... |
| No. 3.... | 818 | 651 | 64 | 3 | 713 | 36 | 5.0 |
| No. 4.... | 816 | 672 | 19 | 3 | 735 | 32 | 4.4 |
| No. 5.... | 750 | 582 | 51 | 6[b] | 683 | 37 | 5.5 |
| No. 6.... | ... | ... | ... | ... | ... | ... | ... |
| No. 7.... | 767 | 602 | 19 | 4[c] | 700 | 48 | 6.9 |
| No. 8.... | ... | ... | ... | ... | ... | ... | ... |

[a] Computed by grouping of observations into classes.

[b] Six values per group except, nine groups of 4 per group and two groups of 3 per group.

[c] Four values per group except, six groups of 3 per group, and four groups of 5 per group.

The computed percentages were compared with those determined from chart 14 of the Committee E-11 publication[3] which is based on the Normal Law Integral. The comparisons are shown in Table V. The differences between the actual and the theoretical percentages shown in this table are probably due both to sampling and construction and testing procedures.

From Table V one can see that the actual percentages tend to approximate closely the theoretical percentages for different values of $t$ equal to or greater than 2. For values of $t$ less than 2, there is fairly good agreement in the case of projects Nos. 1, 4, 6, and 7.

TABLE V.—COMPARISONS OF ACTUAL WITH THEORETICAL PERCENTAGES OF OBSERVATIONS WITHIN THE RANGE, $\bar{X} \pm t\sigma$.

| Range ($R$) ($\bar{X} \pm t\sigma$) | Theoretical Per Cent of Total Observations Lying Within Given Range | Actual Per Cent of Observations Lying in Range $\bar{X} \pm t\sigma$ | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Project No. 1 | Project No. 2 | Project No. 3 | Project No. 4 | Project No. 5 | Project No. 6 | Project No. 7 | Project No. 8 |
| $\bar{X} \pm 1.0\sigma$ | 68.3 | 64.2 | 80.0 | 88.0 | 70.0 | 82.0 | 61.8 | 71.0 | 85.7 |
| $\bar{X} \pm 1.5\sigma$ | 86.6 | 81.2 | 92.0 | 97.7 | 89.0 | 93.0 | 91.0 | 92.0 | 93.3 |
| $\bar{X} \pm 2.0\sigma$ | 95.5 | 92.2 | 97.5 | 99.5 | 93.0 | 98.0 | 97.5 | 98.8 | 96.7 |
| $\bar{X} \pm 2.5\sigma$ | 98.7 | 98.3 | 99.0 | 100.0 | 98.3 | 99.0 | 99.5 | 99.5 | 99.5 |
| $\bar{X} \pm 3.0\sigma$ | 99.7 | 98.3 | 100.0 | 100.0 | 98.5 | 99.9 | 100.0 | 100.0 | 100.0 |

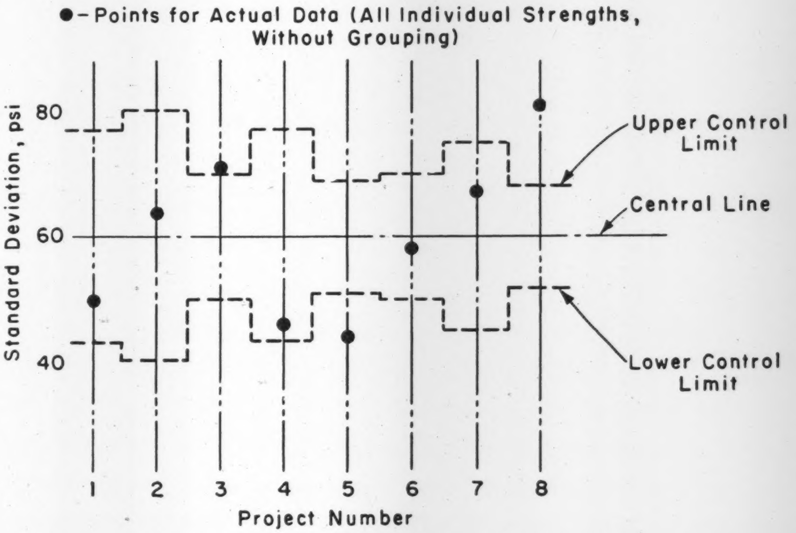

FIG. 3.—Control Chart—Standard Deviation. All Individual Beams (without grouping), 28-Day Flexural Strength Tests.

A statistical analysis of the standard deviations of the eight projects has shown that there is no single theoretical standard deviation, common to all projects. This is shown graphically by Fig. 3 which is a control chart.

Figure 3 is a statistical control chart for the standard deviations of all the individual beams for the eight projects using a mean value of 61 psi. The control chart provides a criterion for detecting lack of statistical control; lack of statistical control is indicated by the fact that observed variations are greater than those due to chance alone. The central line of the chart is the general average of the standard deviation for all the individual beams for all the eight projects. The upper and lower control limits were computed from Eq 4 using a value for $t$ of 3.

It is apparent from Fig. 3 that a standard deviation of about 61 psi is consistent. On projects Nos. 1, 2, 3, 4, 6, and 7 the quality of the concrete was consistent with the degree of field control exercised. On project No. 5 the field control,

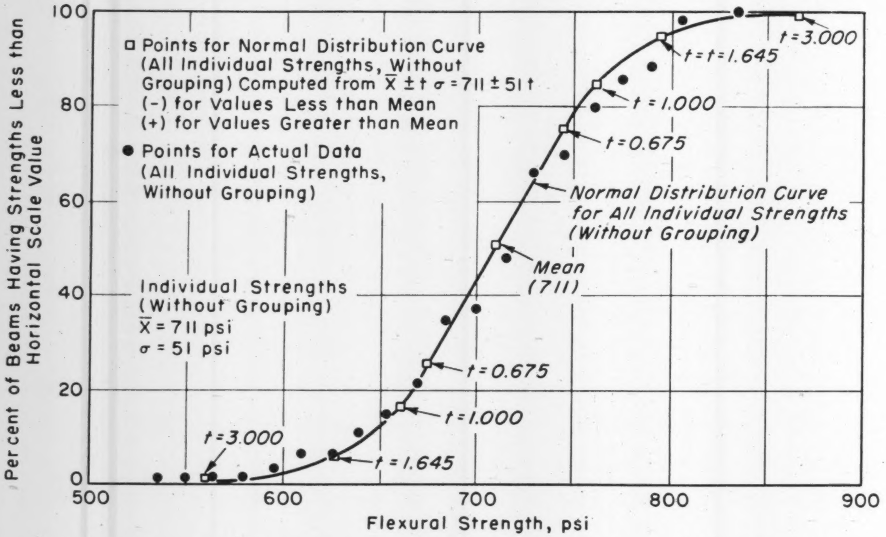

FIG. 4.—Cumulative Frequency Curves—28-Day Flexural Strength. Project No. 1.

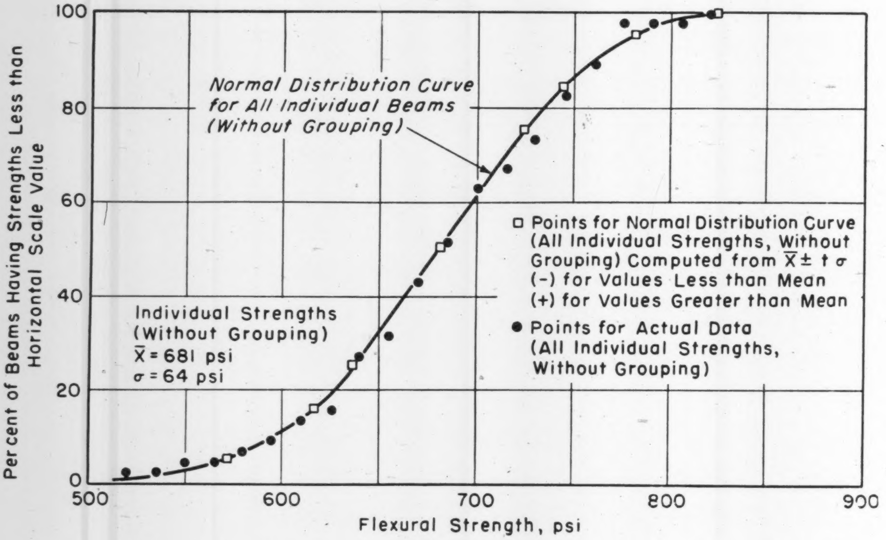

FIG. 5.—Cumulative Frequency Curve—28-Day Flexural Strength. Project No. 2.

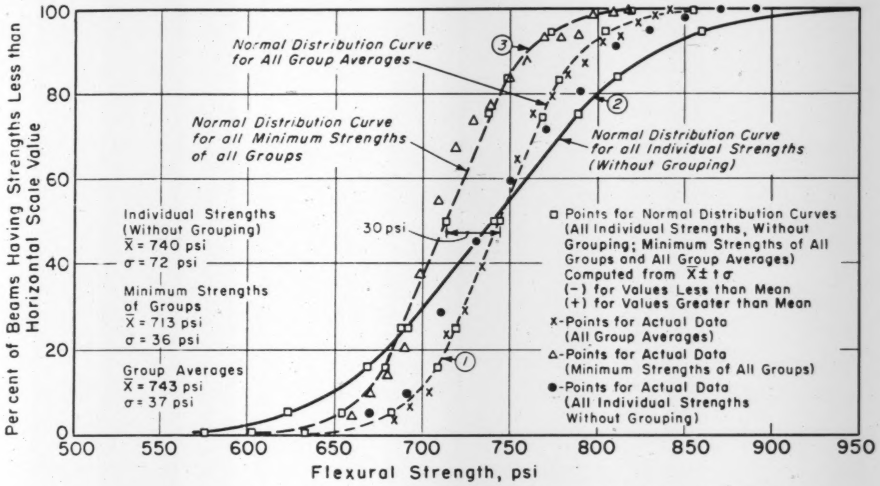

FIG. 6.—Cumulative Frequency Curves—28-Day Flexural Strength. Project No. 3.

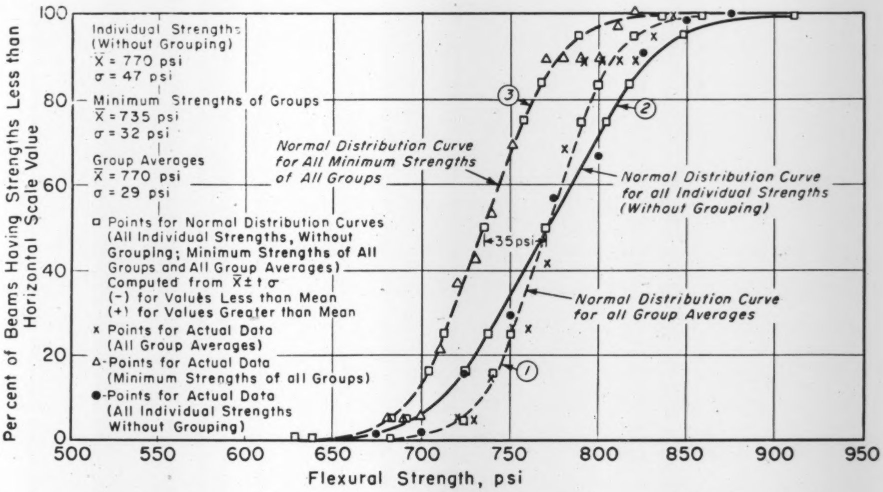

FIG. 7.—Cumulative Frequency Curves—28-Day Flexural Strength. Project No. 4.

according to Fig. 3, was better than the average field control for all eight of the projects. On project No. 8 the degree of field control was poorer than the average for the eight projects. Some cause other than chance, such as poor or soft aggregate, was probably accountable in this case for a lower degree of field control than the average for the eight projects. For a given project, the objective is to have a uniform quality project of high level.

Cumulative frequency curves are shown in Figs. 4 to 12, inclusive. These graphs are explained as follows. In Fig. 4, for example, the full curve is plotted by using Eq 5 and substituting therein the mean value, 711 psi, for $\bar{X}$ and 51 psi for $\sigma$, which are the values shown in Table II for project No. 1. The mean value, 711 psi, is plotted in the figure at the 50 per cent mark on the ordinate scale. At this point, the value for $t$ in Eq 5 is zero. Now by applying the nor-

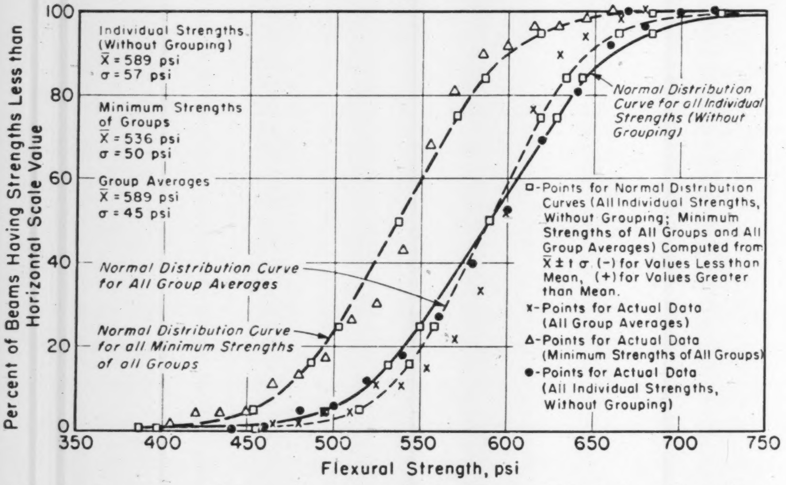

FIG. 8.—Cumulative Frequency Curves—7-Day Flexural Strength. Project No. 5.

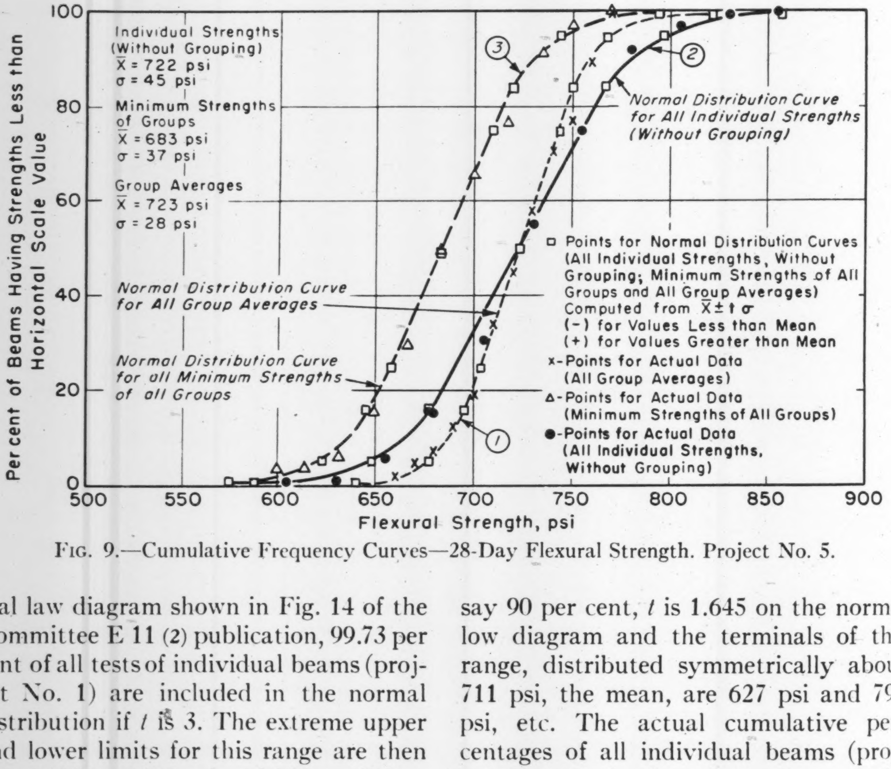

FIG. 9.—Cumulative Frequency Curves—28-Day Flexural Strength. Project No. 5.

mal law diagram shown in Fig. 14 of the Committee E 11 (2) publication, 99.73 per cent of all tests of individual beams (project No. 1) are included in the normal distribution if $t$ is 3. The extreme upper and lower limits for this range are then 711 + 153 = 864 psi and 711 − 153 = 558 psi, respectively, and these values are plotted (squares in Fig. 4) at the terminals of the curve. For a lesser range, say 90 per cent, $t$ is 1.645 on the normal low diagram and the terminals of this range, distributed symmetrically about 711 psi, the mean, are 627 psi and 795 psi, etc. The actual cumulative percentages of all individual beams (project No. 1) having flexural strengths equal to or less than the corresponding value on the abscissa scale are plotted as full circles in Fig. 4. The fact that

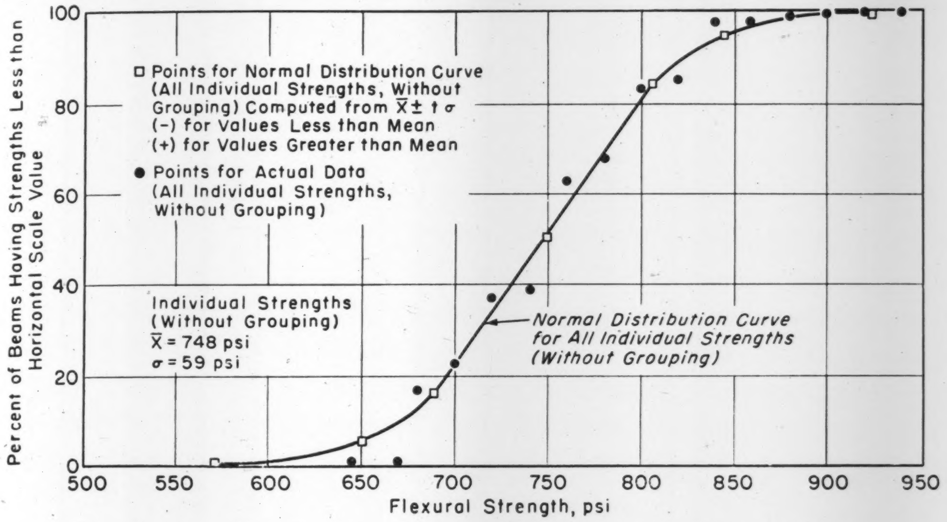

FIG. 10.—Cumulative Frequency Curve—28-Day Flexural Strength. Project No. 6.

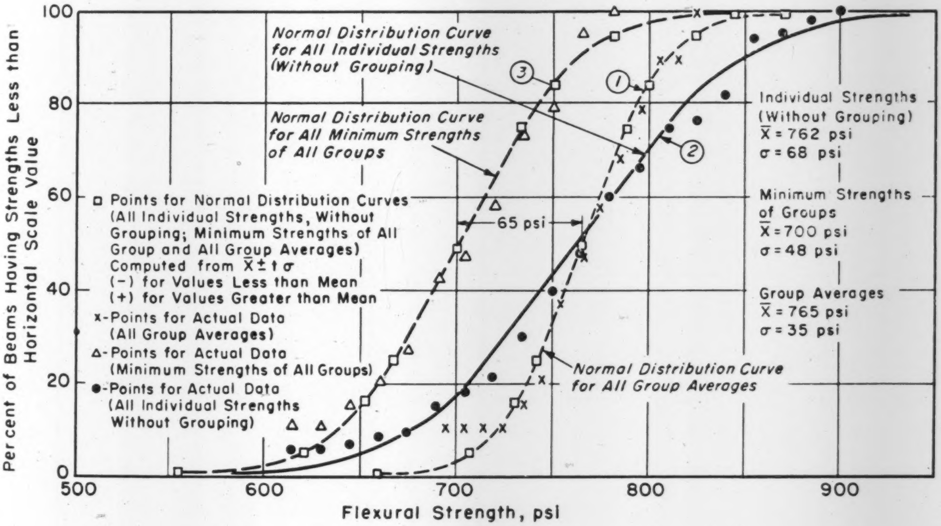

FIG. 11.—Cumulative Frequency Curves—28-Day Flexural Strength. Project No. 7.

these actual values tend to either fall on or close to the normal distribution curve is quite significant, indicating that the data are quite amenable to statistical analysis and that the methods of statistical control are applicable.

The actual data for individual beam tests for project No. 2 are plotted in Fig. 5 and again they tend to be close to the normal distribution curve. Figure 6 shows three normal distribution curves for project No. 3; curve *1* for all group averages,

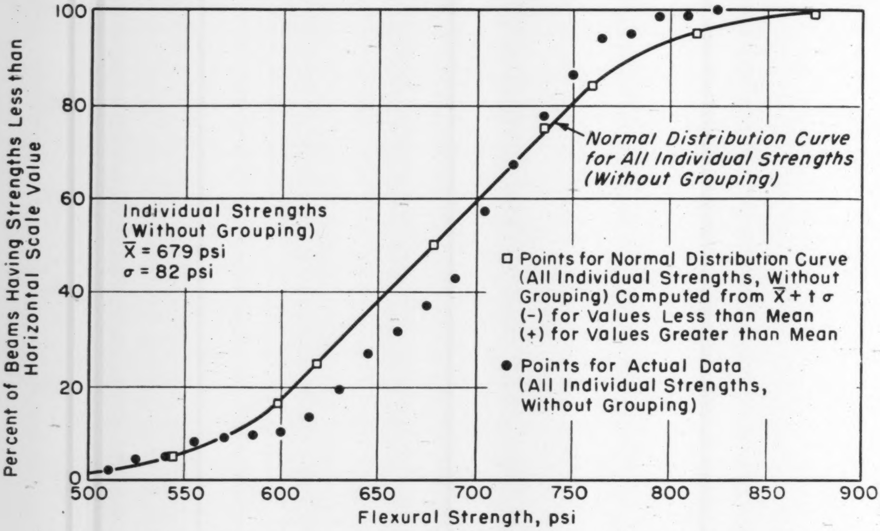

FIG. 12.—Cumulative Frequency Curve—28-Day Flexural Strength. Project No. 8.

curve *2* for individual beams (no grouping of values) and curve *3* for all individual minimum values in all of the groups. In all three cases the actual data when plotted fall either on or close to the normal distribution curves. The same is true for project No. 4, shown in Fig. 7. Figures 8 and 9 (project No. 5) show the cumulative frequency curves at 7 days and at 28 days, respectively, and indicate closest agreement at the 28-day period. Figure 10 (project No. 6) is very similar to the curves of Figs. 4 and 5. Figure 11 exhibits the same pattern as Figs. 6 and 7. These were projects that were widely separated, all having different construction contractors and different testing laboratories in charge of the construction control tests. Figure 12 (project No. 8) shows relatively more deviation of actual data from the normal distribution curve than any of the other projects.

Figure 8 (project No. 5) is very typical of normal distribution or cumulative frequencies at 7 days wet curing of

TABLE VI.—MEAN VALUES OF RATIOS: 7- TO 28-DAY FLEXURAL STRENGTHS.

| Project | Ratios for All Individual Beams[a] | Ratios for All Minimum Values, All Groups[b] | Ratios for All Group, Averages[c] | Values of σ, per cent | | |
|---|---|---|---|---|---|---|
| | | | | *a* | *b* | *c* |
| No. 1.... | 83.1 | ... | ... | 8.4 | ... | ... |
| No. 2.... | 79.5 | ... | ... | 7.6 | ... | ... |
| No. 3.... | 74.5 | 74.0 | 74.1 | 7.0 | 6.4 | 5.3 |
| No. 4.... | 79.3 | 81.6 | 80.9 | 4.3 | 4.3 | 3.7 |
| No. 5.... | 81.4 | 78.8 | 81.3 | 6.2 | 5.3 | 4.0 |
| No. 6.... | 79.3 | ... | ... | 6.9 | ... | ... |
| No. 7.... | 81.0 | 80.9 | 81.5 | 7.3 | 7.9 | 4.1 |
| No. 8.... | 76.5 | ... | ... | 6.9 | ... | ... |

[a] All individual beams.

[b] For all minimum values of all groups.

[c] For all group averages. Per cent.

TABLE VII.—MEAN RATIOS OF 7- TO 28-DAY COMPRESSIVE STRENGTH FOR INDIVIDUAL CYLINDERS.

| Project | Number of Cylinders | Compressive Strength at 7 days, psi | Compressive Strength at 28 days, psi | Ratio, per cent | Standard Deviation, per cent |
|---|---|---|---|---|---|
| No. 5..... | 240 | 4291 | 6044 | 71.0 | 7.9 |
| No. 8..... | 66 | 3107 | 4885 | 63.6 | 8.0 |

beams. Similar curves for the other seven projects are therefore not shown.

### *Ratio of 7- to 28-day Flexural Strength:*

A comparison of 7-day to 28-day flexural strengths is shown in Table VI which is a summary of the ratios.

The averages for the 7- to 28-day ratios were about the same for individual beams, group averages, and individual minimum values within groups. On the average, the ratio in all cases tends to be about 79 per cent, with an extreme range of 74 to 83 per cent. Standard deviations ranged from 4 to 8.4 per cent (see Table VI).

Table VII shows similar data obtained with concrete cylinders, tested in compression at 7 and 28 days. Here the ratio was somewhat lower than those shown in Table VI. The standard deviations for the compressive strength 7- to 28-day ratios are comparable to those of Table VI. This comparison could be improved, however, by having more compressive strength data. In general, the project specifications have required only tests in flexure. The laboratories went beyond this requirement in projects Nos. 5 and 6.

In actual construction acceptance of concrete on the project, in Navy airfield paving, has necessarily been based on flexural strength tests made after 7 days of wet curing. The design of pavement thickness, however, must have reference to the anticipated flexural strength attained at not less than 28 days. Usually, the pavements have actually cured for 60 days or more before they have been subjected to plane traffic.

## DISCUSSION

Each group average was representative of 400 cu yd of concrete. The individual beams were not representative of any specific amount of concrete. The general requirement for testing groups of beams in all projects was only gradually established.

The tests of individual beams have yielded cumulative frequency curves having a greater range than either the curves for group averages or those for the individual minimum values of groups. Obviously, the reduced range realized by testing groups of beams is conducive to better control of flexural strength.

With reference to Figs. 6, 7, 9, and 11 for projects Nos. 3, 4, 5, and 7, respectively, it is noted that the differences between the means for group averages and for minimum values within groups are 30, 35, 40, and 65 psi for the four projects. During actual construction, the maximum difference between a specified group average and the minimum value for any beam in a group seldom exceeded 100 psi. Thus a requirement that a minimum average flexural strength of say 700 psi at 28 days for a group of 3 or more beams with a minimum value of 600 psi for any beam of the group would impose no severe penalty on the contractor *provided* that the divergence at midpoints on the ordinate between the normal distribution curves for minimum values and group averages closely approximates those shown in Figs. 6, 7, 9, and 11. Thus statistical studies and the plotting of normal distribution curves should become extremely useful in the writing of project specifications. They should have a twofold purpose, namely, to obtain a minimum flexural strength for design purposes without inviting overly high bids through concern on the part of contractors that values above the specified minimum for single beams can be attained only by developing group average flexural strengths far above the specified minimum for a group. Again, when cumulative frequency curves can be plotted from a great deal of data, it is possible to estimate the relative frequency of values below the specified minimum group average and the specified minimum individual values of groups when the mix is designed to yield the mean value for the group average. For example, for project No. 4 (Fig. 7), the mean group average is 770 psi. If the mix is designed to yield this value at 28 days, then if the specifications require a minimum group average of 700 psi, about 2 per cent of all groups can be expected to be below 700 psi and if the minimum value for any individual beam of groups is required to be 600 psi, then the expectancy (from Fig. 7) is that no beam will have a flexural strength below this value. The producer who may not be versatile in methods of statistics can use curves such as these without knowledge of the significance of standard deviations used in plotting them.

Page 68 of Bloem's publication[5] gives values of 6 to 23.6 per cent for the coefficient of variation for compressive strength at 28 days. These values have reference to 20 different projects. Again on page 69 of this same publication, for 10 projects, all by the same producer, $v$ ranged from 11 to 21.8 per cent. For projects 5 and 8 of the present paper, $v$ for tests in compression at 28 days is 9.3 and 14.8 respectively, whereas the corresponding $v$ for flexural strengths is 6.2 and 12.0 at 28 days. The values for $v$ for group averages and individual beams as shown in Tables II, III and IV of this paper range from 3.8 to 12.0 per cent. Table I of Himsworth's paper[4] shows values of $v$ ranging from 9 to 32 per cent.

Thus, the data indicate that the coefficient of variation tends to be no greater for tests in flexure than for tests in compression. In Navy pavement construction, no reliable relationship between flexural and compressive strengths has been found to be applicable. Hence, present guide specifications require only tests in flexure. Since the construction of military airfields is worldwide, there is the additional advantage of using very portable equipment in making tests in flexure.

No test data beyond those obtained at 28 days have been presented in this report. However, flexural strength of concrete pavement over a period of years is a consideration of major importance in Navy construction because of the trend to increase the tire loads of naval aircraft. The concrete pavements at more than a hundred naval airfields have been evaluated during the past twelve years. This evaluation includes the cutting out of square sections, sawing them into beams and testing the beams in flexure. Some of the beams of slabs more than 6 years old have had flexural strengths exceeding 1000 psi. Others have had low flexural strengths (400 to 600 psi). These differences cannot be accounted for with any degree of certainty. A general observation, however, seems to be that concrete pavement having good (600 to 800 psi) flexural strength at 28 days tends, with certain exceptions, to increase more in flexural strength with time than slabs having lower flexural strengths at 28 days. The cost of constructing new overlay concrete pavements atop older ones to increase the load capacity when new types of planes require this is such as to warrant some effort to reduce such cost by having a high standard of quality in the mix design. Overdesigning the slab thickness to meet probable future requirements that may never be realized is not practicable from any fiscal standpoint.

## CONCLUSIONS

On the basis of the data presented, the following conclusions seem to be warranted:

1. To obtain concrete airfield pavement of uniformly good or desirable

flexural strength it would be well to specify two control limits—a minimum group average and a minimum value for any individual beam of a group at 28 days wet curing.

2. The mean of the group averages for the projects described tended to be from 7 to 15 per cent higher than the mean for the minimum values of all groups of a project.

3. It seems apparent that it should be advantageous for paving contractors and others to use cumulative frequency curves, obtained with sufficient data, in order to "set a target" for a mean group average flexural strength which, if attained, will tend to minimize without undue cost the number of instances of failure to meet specification requirements with respect to acceptable limits for minimum group averages and minimum values for any individual beam of a group.

4. Considering all projects and data for both group averages and individual beams the average ratio of 7- to 28-day flexural strength was approximately 79 per cent. The standard deviation for this ratio for all tests of individual beams (without grouping) of the 8 projects ranged from 4.3 to 8.4 per cent. The range was from 3.7 to 5.3 per cent for group averages (4 projects) and from 4.3 to 7.9 per cent for the minimum values within these groups. For projects Nos. 5 and 8 the standard deviation for the ratio of strength at 7 days to strength at 28 days was about the same for flexural as for compressive strength. This comparison has reference to tests of individual beams and cylinders without grouping.

5. From the data presented in this paper and from previously published data, it is concluded that the coefficient of variation for flexural strength of beams at 28 days does not exceed that for compressive strength of cylinders at 28 days (in fact, this is some indication that it actually tends to be less).

6. Since the present study as completed thus far has indicated that test data obtained by testing concrete beams in flexure are fully as concordant as data obtained by compression tests of cylinders, it is concluded that it is in the interest of good design and construction to rely on actual tests of concrete beams in flexure than to attempt to estimate the flexural strength from data obtained by testing concrete cylinders in compression.

*Acknowledgments:*

The author is indebted to technical personnel and to the District Public Works Officer of the Sixth U. S. Naval District for collecting and assembling the data. L. A. Palmer, Engineering Consultant in Soil Mechanics and Paving of the Bureau of Yards and Docks, sponsored the investigation, of which this report is a part, and has provided technical guidance during its progress. Philip P. Brown, with whom the author is associated, has given many helpful suggestions.

## DISCUSSION

Mr. Delmar L. Bloem.[1]—One of the conclusions of this paper is that a flexural strength specification should include a minimum strength limit for an individual beam. However, on the curves there were always some beams which tested below this so-called minimum value. In that connection it was stated in presentation that the number of low tests "amounted to small potatoes in relation to the total number of specimens."

Is there some quantitative measure of "small potatoes"? In other words, is it permissible to have 5 per cent of specimens or 1 per cent, or just what per cent below the so-called absolute minimum value?

Mr. L. A. Palmer[2] (*for the author*).—There are various ways of dealing with the problem. One procedure is to have 4 by 4-ft sections cut from the concrete pavement and have beams for flexural tests sawed from the sections. This may be done at 28 days, 60 days, etc., after the concrete is poured. Then if the test data indicate a consistent gain in flexural strength with time and if the number of beams having flexural strengths much below the required minimum is small, the construction is considered as satisfactory. It very rarely happens that the in-place flexural strength of the concrete is so low that the contractor is required, by the terms of the contract, to remove and replace any extensive portion of the pavement.

If a reasonable minimum is set in the project specifications and a contractor does not meet the required minimum in a few instances, it is no calamity. Control tests during construction enable him to find and evaluate the factors contributing to any sudden trend to low flexural strength soon enough to enable him to correct the conditions. Also, the project requires the exercise of sound judgment and common sense. The objective is to correct conditions in time, and a cooperative contractor seldom needs to be penalized. If from 3 to 5 per cent of his concrete is below the absolute minimum in flexural strength, this is not bad at all. For in this case he is producing essentially a good quality of concrete and I would recommend adhering to the standard of a 600-psi minimum and a 700-psi minimum average rather than to increase the range from a 550-psi minimum to a 700-psi group average to eliminate the possibility of having any beams below the allowable minimum in strength.

The whole point is this: when the contractors found out from experience and from study of the data that they could meet the requirements within reason, their confidence was reflected in a tendency to lower bids in all subsequent paving projects. All concerned had to learn that a few below minimum strength values can be due to the human equation in testing as well as to faults of construction procedures. A statistical study helps contractors to estimate how high they need to "raise their sights" to be reasonably certain that they can meet all requirements within reason.

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[1] Associate Director of Engineering, National Sand and Gravel Association, National Ready Mixed Concrete Association, Washington, D. C.

[2] Navy Department, Bureau of Yards and Docks, Washington, D. C.

MR. BLOEM.—The reason I asked the question was that the data have done an excellent job of showing the conformance of the strength tests to normal distribution. Values of standard deviation are shown in the range of approximately 45 to 82 psi. This means that, even if a minimum requirement is disregarded to the extent of permitting 1 per cent of failures, concrete with a required "minimum" of 600 psi would have to have an average strength of as much as 790 psi. Keeping in mind that even such astoundingly large overdesigns will not insure all tests above the minimum, we see the great lack of economy of attempting to enforce an absolute minimum which, as demonstrated by the data, is virtually impossible of attainment. A realistic specification should state the required strength and also indicate specifically what percentage of individual tests may fall below that value to allow for normal variation of testing as well as of the concrete itself.

# RECENT DISINTEGRATION OF MORTAR IN BRICK WALLS*

By C. C. Connor[1] and W. E. Okerson[2]

## Synopsis

Sixty-four brick masonry buildings located in a seacoast state, and constructed from 1945 to 1954 inclusive, were studied in detail to determine the causes of a type of mortar disintegration which had caused widespread damage since 1944. Fifty-one buildings had some depth of disintegration, but 13 had none. In addition, 19 other buildings in nearby areas were inspected. Eight of these showed mortar breakdown, and 11 had none. It was indicated that the mortar disintegration was caused by efflorescence containing sodium and potassium sulfates. The source of the sodium and potassium in these destructive sulfates apparently was the alkali content of the cements used. As far as could be determined, mortars containing cement having more than 0.6 per cent of sodium and potassium oxides (soda equivalent) were consistently associated with disintegration; but where the cement had a lower alkali content, disintegration either did not occur, or with one exception, was only slight. Brick with heavy sanding tended to increase the mortar disintegration, but rough surfaced brick reduced it. Good workmanship probably tended to reduce the mortar disintegration, but it was not a controlling factor. Temperature of the bricklaying period, double tooling, and the sands and limes used, exerted no evident influence. The mortar disintegration was found to be progressive with time.

The disintegration of the face of mortar joints has become a serious problem in many buildings constructed since 1944. The mortar disintegration and the amount of efflorescence associated with it produced a considerable disfigurement, and the openings formed by the disintegration are a threat to the moistureproofness of the buildings. At one building, the disintegration caused leaks three years after construction, requiring expensive moistureproofing treatment. The depth of disintegration at four buildings was $\frac{3}{8}$ in. or more in spots, and a total of 28 buildings showed severe or very severe disintegration. The depth and extent of disintegration grow with time, and these buildings are very likely to require extensive moistureproofing sometime in the future.

### *Purpose of the Investigation:*

The purpose of this investigation was to study a considerable number of brick buildings constructed during the period when this particular type of mortar disintegration occurred, and, if possible to determine the following:

1. The extent and seriousness of the mortar disintegration.

---

* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Consultant, Masonry Construction and Maintenance, Verona, N. J.

[2] Engineer, New Jersey Bell Telephone Co., Newark, N. J.

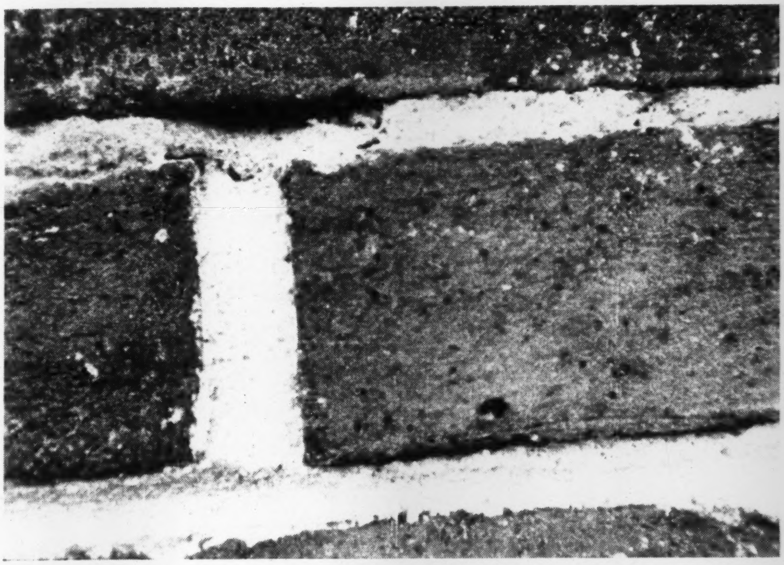

FIG. 1.—Deep Mortar Disintegration and Edge Effects at Building No. 43.

2. The factors that tended to decrease or increase mortar joint disintegration.
3. The materials and practices which exerted no influence on mortar joint disintegration.
4. The combination of factors, if any, which produced resistance to mortar disintegration.
5. The fundamental cause of the mortar disintegration.

*The Condition:*

The type of disintegration of mortar joints considered in this paper affects the surface. In its initial stage it may produce a V-shape groove in the center of the joint, carve out a groove at one or both sides of the mortar joint, or remove the whole surface by spalling off flakes or small particles. As the condition progresses, the whole surface of the mortar joint is spalled off leaving a rough and somewhat pitted surface with the coarse particles of sand exposed.

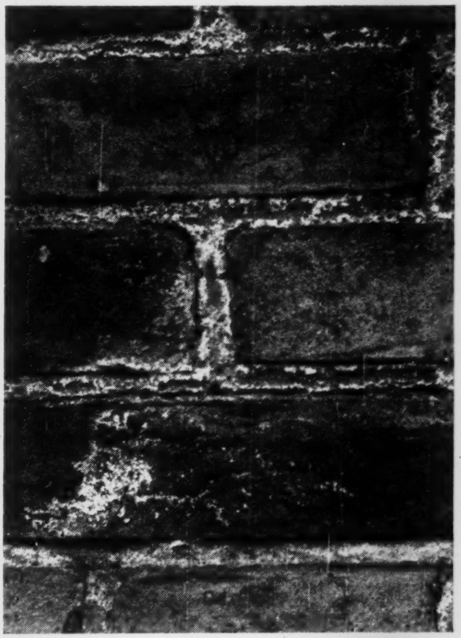

FIG. 2.—Mortar Edge Disintegration. Note Concentration of Efflorescence on Joints. Building No. 3.

TABLE I.—BUILDINGS WITH

| Building | Building Age, yrs | Bricklaying Period | Conditions At Time of Inspection | | | | | | Mortar Joint Disintegration | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | First Inspection Date | Evidence of Active Disintegration? | Conditions Favorable for Efflorescence? | Is Efflorescence Present with Disintegration? | Temperature Between Last Rainfall and Inspection[a] | | Worst Wall Condition | Depth Worst Wall | Distribution Worst Wall |
| | | | | | | | Min | Max | | | |
| No. 1.... | 8 | July to Oct. | 6/14/56 | No | No | ? | ? | ? | Very severe | Very deep | Extensive |
| No. 2.... | 5 | Mar. to June | 6/14/56 | No | No | No | ? | ? | Slight | Shallow | Spotty |
| No. 3.... | 6 | Apr. to July | 3/21/56 | Yes | Yes | Yes | 22 | 47 | Severe | Deep | Extensive |
| No. 4.... | 3 | Aug. to Sept. | 2/14/56 | Yes | Yes | Yes | 28 | 52 | Very slight | Shallow | Scattered |
| No. 5.... | 2 | Sept. to Nov. | 2/24/56 | Yes | Yes | Yes | 14 | 49 | Severe | Shallow | Extensive |
| No. 6.... | 4 | May to July | 10/9/56 | No | No | ? | ? | ? | Slight | Shallow | Spotty |
| No. 7.... | 5 | Apr. to June | 2/24/56 | Yes | Yes | Yes | 14 | 49 | Slight | Shallow | Spotty |
| No. 8.... | 9 | Nov. to Feb. | 10/12/56 | No | No | ? | ? | ? | Very severe | Very deep | Extensive |
| No. 9.... | 8 | Feb. to Apr. | 10/5/56 | No | No | ? | ? | ? | Severe | Deep | Extensive |
| No. 10.... | 5 | Aug. to Dec. | 6/26/56 | No | No | ? | ? | ? | Slight | Shallow | Spotty |
| No. 11.... | 7 | Oct. to Nov. | 6/14/56 | No | No | ? | ? | ? | Slight | Shallow | Scattered |
| No. 12.... | 4 | June to July | 6/28/56 | No | No | ? | ? | ? | Very slight | Shallow | Spotty |
| No. 13.... | 8 | Apr. to July | 4/24/56 | Yes | Yes | Yes | 32 | 72 | Severe | Deep | Extensive |
| No. 14.... | 6 | Aug. to Oct. | 2/23/56 | Yes | Yes | Yes | 14 | 49 | Severe | Deep | Extensive |
| No. 15.... | 4 | ? | 5/2/56 | Yes | Yes | Yes | 39 | 83 | Severe | Deep | Extensive |
| No. 16.... | 5 | Nov. to Jan. | 4/18/56 | No | Yes[i] | ? | 42 | 71 | Severe | Very deep | Extensive |
| No. 17.... | 8 | Sept. to Nov. | 4/6/56 | Yes | Yes | Yes | 33 | 66 | Very severe[k] | Very deep | Extensive |
| No. 18.... | 6 | May to July | 9/26/56 | No | No | ? | ? | ? | Severe | Deep | Extensive |
| No. 19.... | 8 | Mar. to June | 2/23/56 | Yes | Yes | Yes | 14 | 49 | Slight | Shallow | Spotty |
| No. 20.... | 7 | Apr. to June | 9/26/56 | No | No | ? | ? | ? | Severe | Deep | Extensive |
| No. 21.... | 2 | July to Sept. | 8/29/56 | No | No | ? | ? | ? | Very slight | Very shallow | Spotty |
| No. 22.... | 8 | May to June | 2/23/56 | Yes | Yes | Yes | 14 | 49 | Slight | Shallow | Spotty |
| No. 23.... | 9 | Sept. to Nov. | 3/27/56 | Yes | Yes | Yes | 18 | 50 | Very severe | Very deep | Extensive |
| No. 24.... | 4 | ? | 6/26/56 | No | No | ? | ? | ? | Severe | Very deep | Spotty |
| No. 25.... | 4 | July to Oct. | 4/2/56 | Yes | Yes | Yes | 33 | 54 | Moderate | Shallow | Extensive |
| No. 26.... | 3 | Nov. to Feb. | 2/24/56 | Yes | Yes | Yes | 14 | 49 | Severe | Deep | Extensive |
| No. 27.... | 3 | Aug. to Sept. | 10/9/56 | No | No | ? | ? | ? | Severe | Deep | Extensive |
| No. 28.... | 3 | Oct. | 4/6/56 | Yes | Yes | Yes | 33 | 66 | Severe | Shallow | Extensive |
| No. 29.... | 2 | Oct. to Dec. | 2/24/56 | Yes | Yes | Yes | 14 | 49 | Severe | Shallow | Extensive |
| No. 30.... | 2 | Apr. to June | 9/26/56 | No | No | ? | ? | ? | Moderate | Shallow | Extensive |
| No. 31.... | 3 | Oct. to Feb. | 6/13/56 | No | No | ? | ? | ? | Very slight | Shallow | One spot |
| No. 32.... | 8 | June to Aug. | 2/24/56 | Yes | Yes | Yes | 14 | 49 | Very slight | Shallow | One spot |
| No. 33.... | 9 | Nov. to Feb. | 2/23/56 | Yes | Yes | Yes | 14 | 49 | Moderate | Shallow | Extensive |
| No. 34.... | 8 | Oct. to Nov. | 2/23/56 | Yes | Yes | Yes | 14 | 49 | Severe | Deep | Scattered |
| No. 35.... | 5 | Mar. to Apr. | 2/23/56 | Yes | Yes | Yes | 14 | 49 | Severe | Deep | Extensive |
| No. 36.... | 3 | Sept. to Oct. | 4/6/56 | Yes | Yes | Yes | 33 | 66 | Severe | Shallow | Extensive |
| No. 37.... | 8 | Jan. to Mar. | 9/26/56 | No | No | ? | ? | ? | Severe | Very deep | Extensive |
| No. 38.... | 6 | July to Aug. | 3/13/56 | Yes | Yes | Yes | 30 | 57 | Severe | Deep | Extensive |
| No. 39.... | 3 | Aug. to Oct. | 4/2/56 | Yes | Yes | Yes | 33 | 54 | Very slight | Shallow | Spotty |
| No. 40.... | 6 | Mar. to Nov. | 3/15/56 | Yes | Yes | Yes | 31 | 44 | Severe | Deep | Extensive |
| No. 41.... | 9 | July to Aug. | 2/24/56 | No | Yes | ? | 14 | 49 | Slight | Shallow | One spot |
| No. 42.... | 3 | Mar. to June | 4/2/56 | Yes | Yes | Yes | 33 | 54 | Very slight | Shallow | Spotty |
| No. 43.... | 5 | Mar. to May | 4/18/56 | Yes | Yes[i] | Yes | 42 | 71 | Very severe | Very deep | Extensive |
| No. 44.... | 9 | May to Aug. | 4/5/56 | No | Yes | ? | 31 | 68 | Very slight | Shallow | Spotty |
| No. 45.... | 3 | Mar. to Apr. | 4/5/56 | No | Yes | ? | 31 | 68 | Slight | Shallow | Scattered |
| No. 46.... | 3 | May to July | 6/14/56 | No | No | ? | ? | ? | Very slight | Shallow | Two spots |
| No. 47.... | 4 | Dec. to Feb. | 10/5/56 | No | No | ? | ? | ? | Moderate | Shallow | One area |
| No. 48.... | 11 | ? | 4/6/56 | Yes | Yes | Yes | 33 | 66 | Very slight | Shallow | Spotty |
| No. 49.... | 8 | June to Aug. | 4/6/56 | Yes | Yes | Yes | 33 | 66 | Severe | Shallow | Extensive |
| No. 50.... | 3 | May to July | 4/24/56 | Yes | Yes | Yes | 32 | 72 | Severe | Deep | Extensive |
| No. 51.... | 3 | Jan. to Apr. | 4/2/56 | Yes | Yes | Yes | 33 | 54 | Severe | Shallow | Spotty |

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[a] Temperature recorded at nearest reporting weather station. Given only when conditions at inspection were favorable for efflorescence and after rainfall of more than 0.25 in.

[b] Type of lime: SH = Special hydrate.

[c] Brick tested flat side down in 1/8 in. of water for one minute.

[d] Highest test of brick sample judged by precipitate of barium sulfate.

[e] Out of season for efflorescence.

[f] No test record. Average of other tests in same period used.

MORTAR DISINTEGRATION.

| Degree of Efflorescence | | | Mortar Mix and Ingredients | | | | | | Face Brick | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Worst Wall Condition | Distribution Worst Wall Condition | | Mortar Mix | Cement No. | Type of Cement | Lime No. | Type of Lime[b] | Sand No. | Brick No. | Average Rate of Absorption,[c] g in 1 min. | Sulfate Content[d] | Bonding Surface |
| | Joints | Bricks | | | | | | | | | | |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 1 | 1 | 13.6 | Moderate | Smooth |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 2 | 2 | 16.7 | None | Smooth |
| Very heavy | Extensive | Extensive | 1:1:5 | 1 | I | 1 | SH | 3 | 3 | 17.5 | Trace | Heavily sanded |
| Slight | Scattered | Spotty | 1:1:5 | ? | ? | ? | SH | ? | 3 | 15.9 | Trace | Heavily sanded |
| Heavy | Extensive | Extensive | 1:1:5 | 2 | I | ? | SH | 3 | 4 | 4.8 | Heavy | Smooth |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 4 | 3 | 18.6[f] | Trace | Heavily sanded |
| | | | | | | | | | 5[g] | High | Heavy | Smooth |
| Moderate | Spotty | Spotty | 1:1:5 | 1 | I | 2 | SH | 5 | 3 | 18.6[f] | Trace | Heavily sanded |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 6 | 6 | 21.3 | None | Heavily sanded |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 7 | 3 | 10.8 | Trace | Heavily sanded |
| None[e] | None | None | 1:1:5 | 11[h] | I | 2 | SH | 8 | 7 | 17.2 | None | Rough sides only |
| None[e] | None | None | 1:1:5 | 3 | I | 3 | SH | 6 | 3 | 23.5 | Trace | Heavily sanded |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 3 | 8 | 16.9 | Trace | Smooth |
| Heavy | Extensive | Spotty | 1:1:5 | 7 | I | 2 | SH | 9 | 4 | 6.0 | Heavy | Smooth |
| Heavy | Extensive | Spotty | 1:1:5 | ? | ? | ? | SH | 6 | 3 | 16.5 | Trace | Heavily sanded |
| Moderate[i] | Extensive | None | 1:1:5 | ? | ? | ? | SH | ? | 2 | 9.9[f] | ? | Smooth |
| Slight[i] | None | Spotty | 1:1:5 | 4 | I | ? | SH | 10 | 3 | 12.9 | Moderate | Heavily sanded |
| | | | | | | | | | 2[j] | 12.3 | Trace | Smooth |
| Moderate | Extensive | Scattered | 1:1:5 | ? | ? | 2 | SH | ? | 3 | 16.8 | Trace | Heavily sanded |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 11 | 3 | 28.0 | Trace | Heavily sanded |
| Moderate | Extensive | Spotty | 1:1:5 | 2 | I | ? | SH | 6 | 6 | 25.6 | Slight | Heavily sanded |
| None[e] | None | None | 1:1:5 | 3 | I | ? | SH | 11 | 3 | 26.4 | Trace | Heavily sanded |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | ? | 9 | 13.5[f] | None | Lightly sanded |
| Very heavy | Extensive | Extensive | 1:1:5 | 2 | I | 2 | SH | 6 | 6 | 23.2 | Trace | Heavily sanded |
| Heavy | Extensive | Spotty | 1:1:5 | ? | ? | ? | SH | 3 | 6 | 20.8 | None | Heavily sanded |
| None[e] | None | None | 1:2:7½ | ? | ? | ? | SH | 12 | 9 | 13.6 | Trace | Lightly sanded |
| Moderate | Extensive | Spotty | 1:1:5 | 5 | I | 2 | SH | 13 | 3 | 21.6 | Trace | Heavily sanded |
| | | | | | | | | | 10[l] | 34.9 | Trace | Smooth |
| Heavy | Extensive | Extensive | 1:1:5 | ? | ? | 2 | SH | ? | 3 | 21.2[f] | Trace | Heavily sanded |
| | | | | | | | | | 2[j] | 15.4 | None | Smooth |
| None[e] | None | None | 1:1:5 | 1 | I | 3 | SH | 3 | 9 | 17.9 | Trace | Lightly sanded |
| Heavy | Extensive | Spotty | 1:1:5 | 1 | I | ? | SH | 14 | 3 | 22.4 | Trace | Heavily sanded |
| Moderate | Extensive | None | 1:1:5 | 2 | I | ? | SH | 3 | 11 | 8.2 | None | Smooth |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 13 | 9 | 13.7 | Trace | Lightly sanded |
| None[e] | None | None | 1:1:5 | ? | ? | 3 | SH | 12 | 6 | 14.1 | Trace | Heavily sanded |
| Very slight | Spotty | Spotty | 1:1:5 | 6 | I | 2 | SH | 15 | 12 | 14.4 | None | Roughsides & ends |
| Moderate | Extensive | Extensive | 1:1:5 | 2 | I | 2 | SH | 6 | 6 | 21.3 | None | Heavily sanded |
| Heavy | Extensive | Extensive | 1:1:5 | ? | ? | ? | SH | 16 | 6 | 35.7 | Trace | Heavily sanded |
| Heavy | Extensive | Extensive | 1:1:5 | 5 | I | 2 | SH | ? | 6 | 14.3 | None | Heavily sanded |
| Heavy | Extensive | Extensive | 1:1:5 | ? | ? | ? | SH | 17 | 3 | 23.0 | Trace | Heavily sanded |
| None[e] | None | None | 1:1:5 | ? | ? | ? | SH | 10 | 3 | 13.3 | Trace | Heavily sanded |
| Heavy | Extensive | None | 1:1:5 | 5 | I | 2 | SH | 3 | 11 | 9.5 | None | Smooth |
| Heavy | Extensive | Extensive | 1:1:5 | ? | ? | ? | SH | 18 | 3 | 23.0 | Trace | Heavily sanded |
| Heavy | Extensive | Extensive | 1:1:5 | ? | ? | ? | SH | ? | 16[m] | 12.6 | Heavy | Smooth |
| | | | | | | | | | 4 | 12.0 | Heavy | Smooth |
| Heavy | Extensive | Scattered | 1:1:5 | ? | ? | ? | SH | 10 | 6 | 21.7 | None | Heavily sanded |
| Moderate | Spotty | Spotty | 1:1:5 | ? | ? | 2 | SH | 13 | 8 | 13.6 | Trace | Smooth |
| Moderate[i] | Extensive | Spotty | 1:2:7½ | ? | ? | ? | SH | 19 | 3 | 12.9 | Moderate | Heavily sanded |
| Slight | Spotty | None | 1:1:5 | ? | ? | ? | SH | 10 | 11 | 4.3 | Moderate | Smooth |
| Slight | Scattered | None | 1:1:5 | ? | ? | ? | SH | ? | 11 | 6.6 | None | Smooth |
| Very slight[e] | Spotty | Spotty | 1:1:5 | 2 | I | 1 | SH | 9 | 8 | 12.2 | Trace | Smooth |
| Very slight | Scattered | Scattered | 1:1:5 | 7 | I | 2 | SH | 20 | 6 | 14.4[f] | None | Heavily sanded |
| Moderate | Spotty | Spotty | 1:1:5 | ? | ? | ? | SH | ? | 3 | 22.1 | Trace | Heavily sanded |
| Moderate | Extensive | Extensive | 1:1:5 | 8[n] | IA | ? | SH | ? | 3 | 18.9 | Slight | Heavily sanded |
| Heavy | Extensive | Extensive | 1:1:5 | ? | ? | ? | SH | 3 | 3 | 21.2[f] | Trace | Heavily sanded |
| | | | | | | | | | 5[g] | High | Heavy | Smooth |
| Heavy | Extensive | Spotty | 1:1:5 | 9 | I | 2 | SH | ? | 3 | 21.2[f] | Trace | Heavily sanded |
| | | | | | | | | | 2[j] | 15.0 | None | Smooth |

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[g] One common brick wall with returns of brick No. 3.
[h] Mortar very dark. A Hudson Valley cement used.
[i] Conditions unfavorable for maximum efflorescence.
[j] One wall of brick No. 2 with returns of brick No. 3.
[k] Walls very wet during construction.
[l] One wall of brick No. 10 with returns of brick No. 3.
[m] One wall of brick No. 16.
[n] Air entraining cement.

## TABLE II.—BUILDINGS WITHOUT MORTAR DISINTEGRATION.

| Building | Building Age, yrs | Bricklaying Period | Conditions at Time of Inspection | | | | Degree of Efflorescence | | | Mortar Mix and Ingredients | | | | | | Face Brick | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | First Inspection Date | Conditions Favorable for Efflorescence? | Temperature Between Last Rainfall and Inspection[a] | | Worst Wall Condition | Distribution Worst Wall Condition | | Mortar Mix | Cement | Type of Cement | Lime | Type of Lime[b] | Sand | Brick | Average Rate of Absorption[c] g in 1 min | Sulfate Content[d] | Bonding Surface |
| | | | | | Min | Max | | Joints | Bricks | | | | | | | | | | |
| No. 52.... | 6 | Apr. to July | 3/5/56 | Yes | 22 | 65 | None | None | None | 1:1:5 | ? | ? | ? | SH | No. 9 | No. 11[e]<br>No. 14[e] | 7.6<br>18.1 | None<br>Slight | Smooth<br>Rough sides and ends |
| No. 53.... | 9 | May to Aug. | 6/24/56 | No[f] | .. | .. | None[f] | None | None | 1:1:5 | ? | ? | ? | SH | No. 21 | No. 13 | 25.4 | Moderate | Rough sides and ends |
| No. 54.... | 8 | July to Aug. | 2/14/56 | Yes | 28 | 52 | Moderate | Spotty | Spotty | 1:1:5 | ? | ? | ? | SH | No. 6 | No. 12 | 14.9 | Moderate | Rough sides and ends |
| No. 55.... | 8 | Aug. to Oct. | 2/14/56 | Yes | 28 | 52 | Moderate | Spotty | Spotty | 1:1:5 | No. 10 | I | No. 2 | SH | No. 6 | No. 3 | 26.9 | Trace | Heavily sanded |
| No. 56.... | 4 | Apr. to July | 4/2/56 | Yes | 33 | 54 | Very slight | Scattered | None | 1:1:5 | No. 6 | I | No. 1 | SH | ? | No. 9<br>No. 10[h] | 13.5[g]<br>34.9 | Trace<br>Trace | Lightly sanded<br>Smooth |
| No. 57.... | 9 | Apr. to Nov. | 9/26/56 | No[f] | .. | .. | None[f] | None | None | 1:1:5 | ? | ? | ? | SH | ? | No. 13 | 23.1 | Moderate | Rough sides and ends |
| No. 58.... | 2 | Dec. to Feb. | 10/8/56 | No[f] | .. | .. | None | None | None | 1:1:5 | ? | ? | ? | SH | No. 3 | No. 9[i]<br>No. 2[i] | 13.5[g]<br>14.9[g] | Trace<br>None | Lightly sanded<br>Smooth |
| No. 59.... | 3 | Aug. to Dec. | 6/26/56 | No[f] | .. | .. | None[f] | None | None | 1:1:5 | No. 11[j] | I | ? | SH | No. 12 | No. 7 | 14.9 | None | Rough sides only |
| No. 60.... | 2 | July to Sept. | 6/13/56 | No[f] | .. | .. | None[f] | None | None | 1:1:5 | No. 12[j] | I | No. 1 | SH | No. 3 | No. 9 | 13.5[g] | Trace | Lightly sanded |
| No. 61.... | 10 | July to Oct. | 10/12/56 | No | .. | .. | None | None | None | 1:1:5 | ? | ? | ? | SH | No. 6 | No. 14 | 19.6 | Slight | Rough sides and ends |
| No. 62.... | 5 | Nov. to Feb. | 10/5/56 | No | .. | .. | None | None | None | 1:1:5 | ? | ? | ? | SH | No. 3 | No. 15 | 13.5 | None | Rough sides and ends |
| No. 63.... | 2 | June to Aug. | 10/8/56 | No | .. | .. | Very slight | None | Scattered | 1:3 | No. 13[k] | .. | .. | .. | No. 3 | No. 6 | 14.9 | None | Heavily sanded |
| No. 64.... | 3 | Aug. to Nov. | 4/6/56 | Yes | 33 | 66 | Very slight | Scattered | None | 1:1:5 | ? | ? | No. 2 | SH | No. 22 | No. 11<br>No. 17[l] | 8.3<br>5.6 | None<br>None | Smooth<br>Smooth |

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[a] Temperature recorded at nearest reporting weather station. Given only when conditions at inspection were favorable for efflorescence and after rainfall of more than 0.25 in.

[b] Type of lime: SH = special hydrate.

[c] Brick tested flat side down in ⅛ in. of water for one minute.

[d] Highest test of brick sample judged by precipitate of barium sulphate.

[e] One wall only.

[f] Out of season for efflorescence.

[g] No test record. Average of other tests in same period used.

[h] One wall of brick No. 10 with the returns of brick No. 9.

[i] One wall and part of another.

[j] Mortar very dark. A Hudson Valley dark cement used.

[k] Masonry cement.

[l] Trim on walls of brick No. 11.

The depth and extent of the disintegration gradually increase, and the depth may be as much as $\frac{1}{2}$ in. Where small cracks exist between the brick and mortar, the mortar tends to spall away and open up these cracks, and there are many openings thus formed wide enough to receive a knife blade. Such conditions are likely to cause leaks in the walls. Figures 1 and 2 show examples of the mortar disintegration studied.

The type of mortar disintegration under consideration is characterized by intermittent periods of active mortar decay when flakes and small particles of mortar are dislodged from the mortar surface. It was in such periods that the progressive separation of mortar flakes from the surface by efflorescent salts was observed. The periods of active mortar disintegration coincide with the appearance of efflorescence on the mortar joints. In the intervening periods, generally longer and lasting all summer, the mortar disintegration is inactive and there is no evidence of mortar particles being dislodged. The inactive periods continue during the absence of efflorescence and until considerable efflorescence is visible on the joints. There were six buildings, however, where visible efflorescence on the mortar joints did not reactivate a disintegrated mortar condition.

Erosion of mortar joints at buildings constructed prior to 1945 consisted of a dusting action which apparently had no periods of concentrated activity and which did not produce the rough, pitted surface characteristic of the type of disintegration considered in this study, unless the mortar had been frozen. It was characteristic that the older sections of buildings were without evidence of this type of mortar disintegration, while the extensions built since 1944 were frequently affected.

*Definition of Terms:*

*Mortar Disintegration* is used in this paper to describe a condition of mortar joint decay where all or part of the exposed surface is progressively removed to varying depths by the spalling of flakes or particles so that the surface exposed is quite rough and sometimes pitted. The characteristic rough surface distinguishes this type of mortar disintegration from normal erosion of sound mortar.

*Building* is used in this paper to designate any section of a building completed as a unit whether it was the original section or any section added after the original section was completed.

*Efflorescence* is used in this paper to describe a deposit of readily soluble salts, generally white, on the surface of mortar joints or brick. It may be chalky or fluffy or, occasionally, needlelike in appearance.

## BUILDINGS STUDIED

The 64 buildings studied were in a seacoast state and were owned by one company. The study was confined to buildings where construction was started in 1945 to 1954 inclusive, since inspection of many older buildings showed no similar mortar disintegration. Records regarding the masonry materials used were fairly complete, but there were many gaps, especially in the information regarding the brands of cement and lime. The mortars were high-lime mixes except for one masonry cement mortar. In most cases, the brick had a moderate rate of absorption and, as a rule, their soluble sulfate contents were low or nonexistent; but there were a number of exceptions. The buildings studied were a part of a long series where, insofar as practicable, the materials and practices had been selected to obtain moistureproof walls. Fifty-seven had never leaked; at four the leaks had been minor; at three the leaks were severe. The severe leaks at one were obviously caused by mortar disintegration.

Nineteen additional buildings belonging to other companies in neighboring states were inspected for mortar disintegration. In those cases the information regarding the materials used was incomplete and has not been used statistically in the study.

TABLE III.—MORTAR DISINTEGRATION AT BUILDINGS IN AREAS OUTSIDE OF STUDY.

| Location of Buildings | Building | Building Age, yr | Buildings with No Disintegration | Buildings with Disintegration | Mortar Mix | Probable Source of Cement |
|---|---|---|---|---|---|---|
| Philadelphia Area.......... | No. 65 | 4 | x | | 1:1:6 | Lehigh Valley[a] |
| | No. 66 | 8 | x | | 1:1:6 | Lehigh Valley[a] |
| | No. 67 | 4 | | x | 1:1:6 | Lehigh Valley[a] |
| | No. 68 | 4 | | x | 1:1:6 | Lehigh Valley[a] |
| | No. 69 | 5 | x | | 1:1:6 | Lehigh Valley[a] |
| | No. 70 | 4 | x | | 1:1:6 | Lehigh Valley[a] |
| | No. 71 | 2 | x | | 1:1:6 | Lehigh Valley[a] |
| | No. 72 | 7 | x | | 1:1:6 | Lehigh Valley[a] |
| | No. 73 | 2 | x | | 1:1:6 | Lehigh Valley[a] |
| Totals.................... | | | 7 | 2 | | |
| Long Island Area.......... | No. 74 | 4 | | x | 1:1:5 | Lehigh Valley[a] |
| | No. 75 | 4 | | x | 1:1:5 | Lehigh Valley[a] |
| | No. 76 | 2 | | x | 1:1:5 | Lehigh Valley[a] |
| | No. 77 | 2 | | x | 1:1:5 | Lehigh Valley[a] |
| | No. 78 | 3 | | x | 1:1:5 | Lehigh Valley[a] |
| Totals.................... | | | 0 | 5 | | |
| Westchester County Area.... | No. 79 | 2 | x | | 1:3[b] | Hudson Valley |
| | No. 80 | 4 | x | | 1:1:5 | Hudson Valley[c] |
| | No. 81 | 4 | x | | 1:1:5 | Hudson Valley[c] |
| | No. 82 | 5 | x | | ?[d] | Lehigh Valley[a] |
| | No. 83 | 4 | | x | ?[d] | ?[e] |
| Totals.................... | | | 4 | 1 | | |

[a] Shipping area for Lehigh Valley cements by Fair Trades Practices ruling.

[b] Masonry cement mortar containing Hudson Valley cement having low alkali content.

[c] Dark colored mortar. Shipping area for Hudson Valley cements.

[d] A high-lime mortar mix.

[e] Shipping area for both Lehigh Valley and Hudson Valley cements. Light and dark cements used.

## DATA

Tables I and II contain the information obtained in the first thorough inspections of the 64 buildings and information gathered from records. Table I covers the conditions existing at those buildings where there was evidence of mortar disintegration. The ratings given for the degree of mortar disintegration were determined by the depth and extensiveness of the condition on the worst walls. Table II covers the conditions existing at those buildings where there was no mortar disintegration.

Table III covers the conditions observed at buildings of other companies in neighboring states and includes the information which had a high probability of accuracy. The data from Table III have not been used in the statistical interpretations, but provide additional evidence.

## DISCUSSION OF DATA

### *Extent and Seriousness of the Condition:*

Of the 64 buildings in the study, 51 showed mortar joint disintegration of some degree, and 13 showed none. This is a ratio of approximately 4 to 1.

At 28 buildings the disintegration was severe or very severe, which is 44 per cent of the total number of buildings studied. The severity of the condition was rated upon the extensiveness or depth of the disintegration, or both, and was an arbitrary judgment.

The inspections of buildings in other states showed that the incidence of mortar disintegration varies widely. In the Philadelphia area, only two of the nine buildings inspected showed any signs of mortar disintegration. In the Westchester County area only one of the five buildings inspected showed disintegration, but in the Long Island area all five buildings inspected were affected. In general, the conditions in Long Island were more severe than those found at the buildings included in this study.

### *Growth of Mortar Disintegration:*

The ratings of the condition at the buildings in the study show that mortar disintegration is slowly progressive and becomes more serious with time. Notes were made in 1953 concerning the degree of mortar disintegration at 27 of the buildings. The ratings of nine of the 15 buildings which showed some mortar disintegration in 1953 were increased in 1956, and in all probability all have increased in extensiveness or depth of disintegration because those where the rating was not increased in 1956 were classed as severe or very severe in 1953. Five of the 12 buildings without evidence of mortar disintegration in 1953 showed disintegration in 1956. The disintegration seldom showed in less than two years.

### *Influence of Efflorescence on Mortar Disintegration:*

Efflorescence on the face of the mortar joints was evident in all cases where there was active mortar disintegration, that is, where many loose flakes or particles of mortar could be scraped off the joints. It was not feasible to make the inspection of all the buildings in the study during periods when efflorescence was visible, but 35 of those showing disintegration were examined when efflorescence was visible on the brick and mortar. Twenty-nine of these showed active mortar disintegration, with loosened particles, and flakes of mortar apparently being pushed off by an accumulation of efflorescent salts under the flakes. During these inspections, particles and flakes of mortar could be scraped off in a shower. In the six other cases, a slight amount of efflorescence was evident, but no active disintegration. The remaining 16 buildings having mortar disintegration were inspected during the summer months when no efflorescence was evident on the joints, and none of these showed any active disintegration. Several of the buildings which showed active disintegration during periods of efflorescence were reinspected during the summer and the disintegration found to be no longer active.

Among the reinspected buildings were buildings Nos. 3, 5, and 35. Building No. 5 showed a very slight amount of efflorescence on the mortar joints at a second reinspection made on October 17, 1956, and it was observed that this slight efflorescence had reactivated the disintegration and pushed off more mortar flakes and particles. No recent freezing temperatures had occurred. A reinspection was made at building No. 3 on November 20, 1956, three days after a rainfall which had *not* been followed by freezing temperatures, and the mortar disintegration was found to have been reactivated on a low wall which had double exposure. The asphalt joint between the sidewalk and the wall was sprinkled with mortar particles. This wall had been observed three times during the summer, and the disintegration found to be entirely inactive. Building No. 35 was also inspected on November 20 and some active disintegration found, although there had been no signs at inspections during the summer. Figure 3 shows the reactivated mortar disintegration at Building No. 3. Particles of mortar are shown riding on top of the efflorescent formation.

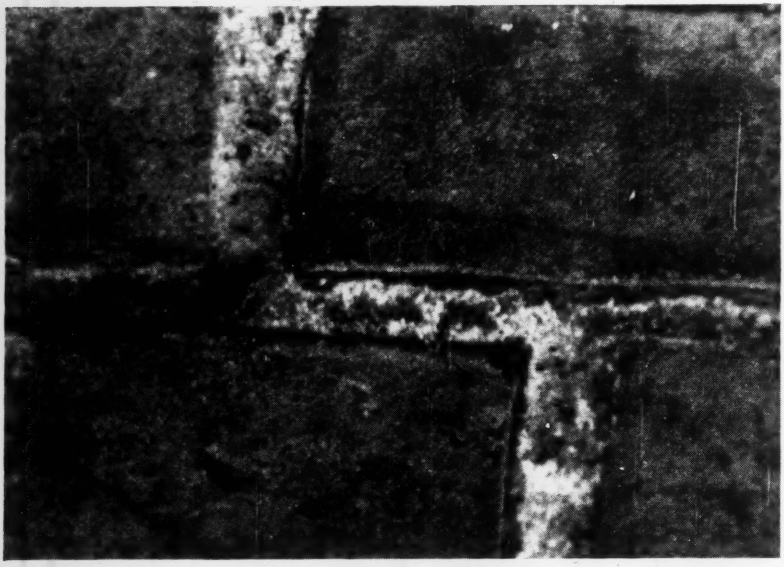

FIG. 3.—Particles of Mortar Riding on Efflorescence on Lower Vertical Joint at Building Nc. 3.

Some observers of this type of mortar disintegration have assumed the cause to be frost action. A confusing factor is that most efflorescence appears during months that have freezing temperatures. However, the efflorescence that reactivated the mortar disintegration at buildings Nos. 3, 5, and 35 during October and November was not accompanied by freezing temperatures, and 11 other buildings with active mortar disintegration were inspected during periods of efflorescence when no frost had followed the previous rainfall. This evidence indicated that frost action was not a primary cause of the disintegration. Furthermore, scores of other buildings, owned by the same company—many of them with similar mortar mixes—had endured frost action for a number of years without showing signs of a similar type of mortar disintegration. It is very doubtful if frost action exerted any important influence.

The evidence was strong that the type of mortar disintegration studied was caused by efflorescent salts which formed at or under the face of the joints and disrupted the mortar.

Efflorescence on mortar joints was not noted to any great extent at this same company's buildings constructed from 1925 to 1945, and then only in a few cases where extensive efflorescence on the bricks had apparently contaminated the mortar joints. In late years efflorescence on mortar joints has been the rule rather than the exception. The first noted appearance of extensive efflorescence on the mortar joints correlates quite well in time with the beginnings of the type of mortar disintegration under consideration:

*Chemical Analysis of Efflorescent Salts:*

A sample of the efflorescent salts was scraped from the surface of the brickwork at building No. 3, and a chemical analysis showed the components listed in Table IV.

TABLE IV.—ANALYSIS OF EFFLORESCENT SALTS.

| Component of Sample | Building No. 3, per cent |
|---|---|
| Total silica as $SiO_2$ | 6.22 |
| Total aluminum as $Al_2O_3$ | 0.91 |
| Total iron as $Fe_2O_3$ | 0.44 |
| Total calcium as CaO | 0.23 |
| Total magnesium as MgO | 0.32 |
| Total sulfur as $SO_3$ | 40.70 |
| Water soluble sodium as $Na_2O$ | 26.96 |
| Water soluble potassium as $K_2O$ | 18.80 |
| Loss on ignition | 5.76 |
| Carbon dioxide, $CO_2$ | 4.22 |
| Water insoluble | 8.24 |

The above analysis was on a "dry basis."

Obviously the greater part of the efflorescence was sodium and potassium sulfates. The disintegrating effect of these salts has been well established. Sodium sulphate is one of the salts used in ASTM method C 88[3] because of its known disintegrating action. It would be logical to conclude that the amount of sodium and potassium sulfates indicated in the analysis of the efflorescent salts would disintegrate the mortar if these sulfates crystallized at or near the surface. Since all of the observations indicated that efflorescence was the mechanical cause of the type of mortar disintegration studied, it is felt that the sodium and potassium sulfates were a primary cause.

Ritchie (**1**)[4] in his study of efflorescence on brickwork piers also found that the major components of the deposit were sodium and potassium sulfates. The samples of efflorescent salts analyzed came from two piers, one of which had a high-lime mortar and the other a straight cement mortar.

*Conditions Favorable to Efflorescence:*

Robertson (**2**) and Ritchie (**1**) have pointed out that efflorescence appears on the surface of brickwork when the drying temperature is low and is probably deposited deep within the brickwork during warm drying weather. The conditions required for the maximum formation of efflorescence on the surface of brickwork are not entirely clear, but U. S. Weather Bureau data given in Tables I and II show that efflorescent salts were deposited on the surface of the buildings only during relatively low temperature periods. A detailed study of the weather data indicated that plentiful efflorescence occurred at the surface under the following weather conditions:

1. An initial soaking rainfall of at least 1 in. in 24 hr. Penetration into masonry is aided by wind, and the precipitation should be spread out over several hours.
2. Favorable drying conditions for two days between rainfalls, or one day with a breeze.

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[3] Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 3.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1188.

The most favorable drying conditions apparently were:

(*a*) Average daily temperature between 35 and 45 F.

(*b*) Maximum daily temperature sufficiently high to melt any ice in the wall and low enough to keep the average temperature below 45 F. Maximum temperatures from 35 F to 55 F were the usual.

NOTE.—Lesser rainfalls following an initial heavy precipitation within a week or so produced plentiful efflorescence; and repeated wetting-and-drying cycles with temperatures favorable to the formation of efflorescence at the surface greatly increased it.

Efflorescence can form at temperatures outside of the ranges given. Spots may form at temperatures well above 50 F, but daily average temperatures above 50 F apparently reduced the amount of efflorescence materially. Several days of continuous freezing immediately after a rainfall dried the moisture without producing visible efflorescence. Apparently the water must be free to move to the surface to deposit visible salts. Relative humidity could not be identified as an important influence, but the daily minimums were generally below 70 per cent. Periods with the required conditions for plentiful formation of efflorescence may be months apart. The conditions in the fall of 1956 were only borderline, and there was no widespread efflorescence.

### *Relation of Brick Laying Periods to Mortar Disintegration:*

Frozen mortar disintegrates in a fashion which resembles the type of disintegration considered in this study, and it is very difficult to differentiate between the two types. The brick laying periods were, therefore, investigated to find if their temperature had any influence on mortar disintegration.

Freezing temperatures can be expected in the months from November to March inclusive, and frozen mortar becomes a possibility if any of the bricks are laid in those months. Table V shows the relation between the bricklaying periods and mortar disintegration. The fact that 26 buildings where the bricks were laid in warm weather had mortar disintegration indicates that frozen mortar at the time of brick laying was not a prime cause.

TABLE V.—RELATION OF TEMPERATURE OF BRICK LAYING PERIOD TO MORTAR DISINTEGRATION.

| Brick Laying Period | Buildings with No Disintegration | Buildings with Disintegration | Totals | Percentage of Buildings with Disintegration |
|---|---|---|---|---|
| Frost period—November to March inclusive | 5 | 22 | 27 | 81 |
| Period without severe frost—April to October inclusive | 8 | 26 | 34 | 76 |
| Totals | 13 | 48 | 61[a] | |

[a] The brick laying periods were known for only 61 of the 64 buildings studied.

### *Influence of Double Tooling on Mortar Disintegration:*

Double tooling of the mortar joints was specified for all the buildings in this study and, in most part, it appears to have been done. Since double tooling produces a compact and dense surface, there was a possibility that this surface might have caused the efflorescent salts to be deposited behind it with a resultant spalling of the mortar.

Double tooling was not specified for

the buildings inspected in other areas. The breakdown of the mortar at five buildings in the Long Island area and at three buildings in the other two areas inspected, as shown in Table III, was good evidence that double tooling was not an important cause of mortar disintegration.

### *Influence of Workmanship on Mortar Disintegration:*

Since mortar disintegration is associated with efflorescence on the mortar joints, it was thought that good workmanship might reduce the efflorescence by reducing joint cracking and keeping water out of the wall. It was recognized however, that there are other causes for mortar joint cracking besides poor workmanship.

Ratings of the quality of workmanship were not always made in this study but were available for a number of the buildings from previous studies. Additional ratings were made during the inspections.

The indications were that good workmanship alone did not prevent efflorescence and mortar disintegration. The workmanship was good or excellent at 15 of the 19 buildings with severe or very severe disintegration, where the workmanship had been judged. At 6 of these the mortar joint cracking was low. The workmanship was good or excellent at 12 of the 13 buildings without mortar disintegration, but the mortar joint cracking was high or very high at two. Other influences appear to have been more important than workmanship.

There were inconclusive indications that poor workmanship may have increased mortar disintegration at some of the buildings in the study, but there were four buildings *without* disintegration in other areas where the workmanship was poor to fair and the joint cracking was high.

It is felt that, in general, good workmanship tended to decrease mortar disintegration, but it was not a controlling factor. Severe mortar disintegration occurred in spite of excellent workmanshp and very low joint cracking.

TABLE VI.—INFLUENCE OF THE MAKE OF BRICK ON MORTAR DISINTEGRATION.

| Brick | Buildings with No Mortar Disintegration | Buildings with Mortar Disintegration | Totals |
|---|---|---|---|
| No. 1...... | | 1 | 1 |
| No. 2...... | | 5 | 6 |
| No. 3...... | 1 | 22 | 23 |
| No. 4...... | | 3 | 3 |
| No. 5...... | 1 | 1 | 2 |
| No. 6...... | 1 | 10 | 11 |
| No. 7...... | 1 | 1 | 2 |
| No. 8...... | | 3[a] | 3 |
| No. 9...... | 3 | 4 | 7 |
| No. 10...... | 1 | 1 | 2 |
| No. 11...... | 2 | 4 | 6 |
| No. 12...... | 1 | 1 | 2 |
| No. 13...... | 2 | | 2 |
| No. 14...... | 2 | | 2 |
| No. 15...... | 1 | | 1 |
| No. 16...... | | 1 | 1 |
| No. 17...... | [Trim only] | | |
| | — | — | — |
| Totals...... | 17 | 57 | 74[b] |

[a] Mortar disintegration very slight in all cases.

[b] Ten buildings had walls of different bricks, making a total of 74 units.

### *Influence of Brick on Mortar Disintegration:*

A study was made of the results obtained with the various makes of brick used in the buildings included in this investigation: There were 17 different kinds of bricks used, but brick No. 17 was used only as a trim on walls at building No. 64 which, in the main, was built with brick No. 11. Table VI shows the results in detail.

It is obvious that bricks Nos. 3 and 6 were associated with mortar disintegration in a large percentage of cases. The wall with common brick No. 5 in building No. 6 had no disintegration, although disintegration was evident on other walls.

*Influence of Brick Bonding Surfaces:*

It was felt that the reason for certain bricks being associated with a high percentage of buildings with mortar disintegration might be the characteristics of their bonding surfaces. Table VII shows the influence of the types of brick bonding surfaces.

TABLE VII.—INFLUENCE OF BRICK BONDING SURFACES ON MORTAR DISINTEGRATION.

| Bonding Surfaces | Brick | Buildings with No Mortar Disintegration | Buildings with Mortar Disintegration | Totals |
|---|---|---|---|---|
| Rough sides and ends | No. 12<br>No. 13<br>No. 14<br>No. 15 | 6 | 1 | 7 |
| Rough sides only | No. 7 | 1 | 1 | 2 |
| Smooth moderate suction | No. 1<br>No. 2<br>No. 4<br>No. 8<br>No. 11<br>No. 16 | 3 | 17 | 20 |
| Smooth high suction | No. 5<br>No. 10 | 2 | 2 | 4 |
| Lightly sanded | No. 9 | 3 | 4 | 7 |
| Heavily sanded | No. 3<br>No. 6 | 2 | 32 | 34 |
| Totals | | 17 | 57 | 74[a] |

[a] Ten buildings had walls of different bricks, making a total of 74 units.

Table VII shows the highly beneficial effect of rough bonding surfaces. Seven of the nine bricks with such surfaces showed no mortar disintegration. Rough bonding surfaces materially reduce the amount of mortar joint cracking if the workmanship is good, and this in turn reduces the amount of water getting into the wall and the amount of efflorescent salts that are leached out. This would tend to reduce the amount of mortar disintegration.

The adverse influence of heavily sanded brick bonding surfaces is obvious with 32 of the 34 such bricks being associated with mortar disintegration. It has been known that heavily sanded brick was a possible hazard to the moistureproofness of buildings because the mortar tends to pull the loose sand from the surface and produce cracks. However, such brick were associated with leaks at only two of the buildings, and there were other adverse factors present. It was noted that an unusual amount of mortar joint cracking had occurred at many of the buildings, and this unusual cracking was, as a rule, associated with the heavily sanded bricks. This cracking would permit the entrance of water and very likely increase the amount of efflorescence if there were efflorescent materials in the mortar.

Two of the rough-surfaced bricks were associated with mortar disintegration; and two of the heavily sanded bricks were in buildings that showed no such evidence, although the joint cracking was high. These exceptions indicate that the brick bonding surface was not the prime cause of mortar disintegration.

TABLE VIII.—INFLUENCE OF SANDS ON MORTAR DISINTEGRATION.

| Sand[a] | Quality of Grading[b] | Buildings with No Mortar Disintegration | Buildings with Mortar Disintegration | Totals |
|---|---|---|---|---|
| No. 3...... | Excellent | 4 | 8 | 12 |
| No. 6...... | Poor | 3 | 6 | 9 |
| No. 9...... | Poor | 1 | 2 | 3 |
| No. 10...... | Good | | 4 | 4 |
| No. 12...... | Good | 1 | 2 | |
| No. 13...... | Excellent | | 3 | 3 |
| Totals...... | | 9 | 25 | 34 |

[a] Sands known to have been used in three or more buildings.

[b] The quality of grading is based upon Connor's recommended grading requirements (**3**).

### *Influence of Sands on Mortar Disintegration:*

The sand used was known at 51 of the 64 buildings studied. There were 22 different sands, but Table VIII shows the relation to mortar disintegration only of those sands which were used in the mortar of three or more buildings.

The sands with good or excellent gradings were associated in 17 cases with mortar disintegration, and the two poorly graded sands were used in four buildings where there was none. Sands from the same pits as Nos. 3, 9, 12, and 13 have been used extensively in concrete with no known indication of being alkali reactive. There was no significant indication that the sands used influenced mortar disintegration.

### *Influence of Limes on Mortar Disintegration:*

There were three different brands of lime used in the buildings studied. All of them were special hydrates.

Table IX shows that limes Nos. 2 and 3 were associated with a high proportion of buildings with mortar disintegration. However, in view of the fact that lime No. 2 and limes in general have very low amounts of soluble alkali to contribute to the alkali salts in the efflorescence, which apparently caused the mortar disintegration, it is felt that the limes used did not exert any significant influence on the disintegration. This subject is discussed later.

TABLE IX.—INFLUENCE OF BRANDS OF LIME ON MORTAR DISINTEGRATION.

| Lime | Type of Lime[a] | Buildings with No Mortar Disintegration | Buildings with Mortar Disintegration | Totals |
|---|---|---|---|---|
| No. 1...... | SH | 2 | 2 | 4 |
| No. 2...... | SH | 2 | 14 | 16 |
| No. 3...... | SH | | 3 | 3 |
| Totals...... | | 4 | 19 | 23[b] |

[a] Type of lime: SH-Special Hydrate.

[b] The brand of lime was known at only 23 buildings of the 64 studied.

To make more certain that the special hydrates were not an influence on mortar disintegration, four buildings constructed in 1942 and one built in 1940, all with 1:1:5 mortars containing specially hydrated lime, were carefully inspected. None of these buildings had any evidence of the type of mortar disintegration under consideration except for a small suspicious-looking spot at one.

### *Influence of Excess Water Entering Walls:*

Several conditions were noted where details of construction shed large amounts of water down over the wall surface, and these were generally accompanied by severe mortar disintegration if the mortar in other areas showed signs of disintegration. Cornices without proper drips, flush copings, slip sills, and corners at set-back walls frequently created concentrated run-downs of water and produced areas of severe disintegration.

TABLE X.—INFLUENCE OF CEMENTS ON MORTAR DISINTEGRATION.

| Cement | Source |  | Soda Equivalent,[a,b] per cent | Sulfur Trioxide,[a] per cent | Tricalcium Aluminate,[a] per cent | Cement Type | Degree of Mortar Disintegration Number of Buildings |  |  |  |
|---|---|---|---|---|---|---|---|---|---|---|
|  | Lehigh Valley | Hudson Valley |  |  |  |  | None | Very Slight or Slight | Moderate to Very Severe | Totals |
| No. 1 | X |  | 0.81 | 2.28 | 13.3 | I |  | 1 | 3 | 4 |
| No. 2 | X |  | 0.3 to 0.8 | 1.9 | 13.6 | I |  | 3 | 3 | 6 |
| No. 3 | X |  | 0.8 | 2.1 | 14.5 | I |  | 1 | 1 | 2 |
| No. 4 | X |  | 0.5 to 0.6 | 2.0 to 2.1 | 10.0 | I |  |  | 1 | 1 |
| No. 5 | X |  | 0.69 | 2.02 | 14.4 | I |  |  | 3 | 3 |
| No. 6 | X |  | 0.6 | 2.4 | 14.0 | I | 1 | 1 |  | 2 |
| No. 7 | X |  | ? | 2.1 | 12.0 | I |  |  | 2 | 2 |
| No. 8[c] |  |  | ? | ? | ? | IA |  |  | 1 | 1 |
| No. 9 | X |  | 0.7 to 0.8 | 2.5 | 13.7 | I |  |  | 1 | 1 |
| No. 10 | X |  | 0.55 to 0.63 | 2.2 to 2.3 | 9.3 | I | 1 |  |  | 1 |
| No. 11 |  | X | Under 0.5 | 1.8 | Under 8.0 | I[d] | 1 | 1 |  | 2 |
| No. 12 |  | X | 0.6 | 1.79 | 5.8 | I[d] | 1 |  |  | 1 |
| No. 13[e] |  |  | ? | ? | ? | [e] | 1 |  |  | 1 |
| Totals |  |  |  |  |  |  | 5 | 7 | 15 | 27[f] |

[a] Typical analyses from manufacturers.

[b] Weight per cent of $Na_2O$ plus 0.658 times weight per cent of $K_2O$.

[c] Air entraining cement.

[d] Also natural type II cement.

[e] Masonry cement with both portland and natural cements.

[f] The brand of portland cement was known at only 26 of the 63 buildings where it was used in a high-lime mortar.

### *Influence of Cements on Mortar Disintegration:*

The brand of portland cement was known at only 26 of the buildings. There was one masonry cement containing unknown percentages of natural and portland cements. Table X gives the source of the cements, a partial analysis where obtainable, and information regarding the mortar disintegration accompanying their use.

It was not possible to obtain chemical analyses of any of the mortars, but several cement manufacturers cooperated and furnished typical analyses of their cements. In most cases it was stated that the content of alkali (sodium and potassium oxides expressed as soda equivalent) varied but little at the same plant, but this was not true of all. The amount of alkali content was not always available.

It should be emphasized that the cements used in the buildings studied were not analyzed and there is no certainty that a typical analysis furnished some years later for a brand of cement would approximately duplicate the analysis of that brand at the time of its use in the buildings studied. Furthermore, the information furnished regarding the alkali content of the cements sometimes covered a considerable percentage range as shown in

Table X. For statistical purposes, cement No. 2 has been considered to have an alkali content of more than 0.6 per cent (soda equivalent) and cement No. 10 to have 0.6 per cent or less. These possible variables unavoidably weaken the force of the statistical evidence concerning the safe limit for the alkali content of cement but it is nevertheless of some interest. For construction purposes an approximate safe limit is much better than none at all.

There were seven buildings studied where cements with an alkali content of approximately 0.6 per cent or less had been used. Four of these buildings had no mortar disintegration, two had slight or very slight amounts, and one was severely affected. All of the 16 buildings with cements containing more than 0.6 per cent alkali had some degree of disintegration, 9 of them severe.

The buildings in outside areas furnished some supporting evidence. All of the available analyses of Hudson Valley cements showed an alkali content of 0.6 per cent or less. The three buildings in the Westchester County area, where the cements were probably from the Hudson Valley, had no disintegration.

An odd situation at building No. 38 seems significant. The brickwork had been completed, using the light-colored cement No. 5 which had a rather high alkali content, when a change in plans required the filling of a good-sized opening with brickwork. The mortar in the patch was very dark, and since this building is in fair territory for both Lehigh Valley and Hudson Valley cements, it seems probable that the cement was a dark Hudson Valley with a low alkali content. Six years later the dark patch had no mortar disintegration but was completely surrounded by brickwork where the disintegration was severe. The condition, at the very least, illustrated the important influence of cements on mortar disintegration.

*Possible Sources of the Alkali Sulfates in Efflorescence:*

The identification of the source of the destructive sodium and potassium sulfates in the efflorescent salts was of prime importance. The presence of sodium, potassium, and sulfur trioxide in cements made them suspect, but other possible sources were considered.

Lime proved to be an unlikely source because of its usually low sulfate content. Typical analyses furnished by the manufacturer of lime No. 1 showed 0.02 to 0.04 per cent of soluble sulfates expressed as $SO_3$. This lime was used at building No. 3 where the efflorescence sample was taken. A typical analysis of lime No. 2 showed 0.03 per cent of $SO_3$. Palmer (4) found that the sulfates in lime were low and mostly calcium sulfates. Ritchie (5) analyzed three limes and found sodium and potassium in two but no $SO_4$. His highest total of sodium and potassium oxides was 0.11 per cent, or 0.09 soda equivalent. All these findings indicate that the lime used at the buildings studied did not contribute materially to the alkali sulfates.

Tests for soluble sulfates were made on samples of several of the sands used, but no more than a trace was ever found. The water used was always potable and the amount of sulfates would be negligible. Several tests showed none.

The approximate soluble sulfate content of the bricks used is given in Tables I and II. In 57 of the 74 tested there were no soluble sulfates, or only a trace. Forty-five of these were in brickwork showing mortar disintegration.

The test for soluble sulfates in brick consisted of soaking 25 g of brick chips from the outer $\frac{1}{4}$ in. of the brick surface in distilled water for 30 min and judging the amount of sulfates by the amount of precipitate thrown down when a few drops of 10 per cent barium chloride solution were added to the filtered water. Brick with no more than a slight amount of sulfates as indicated by this test showed no tendency to effloresce in a long succession of buildings. The elimination of brick with a tendency to effloresce, the elimination of sands containing chlorides, and the use of moistureproof construction practices resulted in 43 efflorescence free buildings in the 7-yr period preceding 1945. The efflorescence at building No. 48, built in 1945, was the first break in the series. Eight kinds of brick were used in these buildings.

Two kinds of brick were used both before 1945 and after. In the 7-yr efflorescence free period preceding 1945, all of the 34 buildings in which brick No. 2 was used showed no efflorescence; but in this study covering buildings constructed in the period from 1945 to to 1954, three buildings out of the four with walls containing brick No. 2 and inspected during conditions favorable for efflorescence had efflorescence on those walls, two on the brick. Brick No. 2 generally tested free of soluble sulfates and never tested to have more than a trace. Brick No. 11 was used in two buildings prior to 1945 and no efflorescence developed, but in the six buildings where it was used after 1944 efflorescence showed on the walls of five. Tests of this brick showed no soluble sulfates except in one case. Brick No. 11 had an impervious fire-flashed face which was a barrier to absorption of water and evaporation. The efflorescence concentrated on the mortar joints and, at four buildings, was accompanied by mortar disintegration.

The brick were not tested for sodium or potassium. It was assumed that if these alkalis were present in soluble form, they would occur as sulfates. Palmer (4) speaks of soluble sulfuric anhydride ($SO_3$) as the chemist's guide to the soluble salts present in face brick. Butterworth (6) writes of the soluble salts in brick that cause efflorescence as being the alkali sulfates and magnesium, calcium, and ferrous sulfates. He further states that the alkali sulfates are decomposed at temperatures below those usual in brick kilns so that the finished product rarely contains more than small amounts.

Apparently the bricks used in the buildings studied were an unlikely source of the sodium or potassium in the alkali sulfates found in the efflorescence. It would not seem probable that the 45 bricks before mentioned with no soluble sulfates or no more than a trace could have been an important source.

In industrial areas there is considerable $SO_3$ in the air, but sodium and potassium are hardly possible. Twenty of the buildings with mortar disintegration were in residential communities where a high content of $SO_3$ in the air would be unlikely.

The virtual elimination of other possible sources of the alkali sulfates found in the efflorescence makes cement, with its known alkali and $SO_3$ content, the most probable. But there was considerable positive evidence.

Ritchie (1) in his research on brick piers found that the main components of the efflorescence developed were sodium and potassium sulfates and that where efflorescence occurred, the amount of efflorescence generally increased with the proportion of the cement in the mortars. Furthermore, he found (5) in his wick tests for efflorescent salts in mortars, that the amount of efflorescence on the wicks was related to the amount of sodium and potassium oxides in the cementing materials; and that, under the same conditions, the lesser amounts of efflorescence were produced by mortars in which the portland cement contained less alkaline material. His chemical analyses showed that the cements used contained considerable amounts of alkaline materials, while the limes had very little. The alkali salts in the efflorescence on the wicks, however, were predominantly carbonates, although some sulfates were present where the mortars contained portland cement. It is not clear why the alkali salts were predominantly carbonates in the efflorescence from one-week old mortar, but were entirely or almost entirely sulfates in the efflorescence from Ritchie's brick piers (1) and from the brick buildings in this study.

A summary of the evidence regarding cement as a possible source of the sodium and potassium sulfates found in the efflorescence on the buildings in this study is as follows:

1. Ritchie (5) found a relation between the amount of efflorescence on clay wicks, the amount of cement in the mortar, and the amount of alkali in the cements, but the alkali salts in the efflorescence were predominantly carbonates.
2. Ritchie (1) found that the main components of the efflorescence on brick piers were sodium and potassium sulfates, and that the efflorescence generally increased with the proportion of cement in the mortar where efflorescence occurred.
3. It was found in this study that the mortar disintegration was evidently caused by efflorescence; that the main components of the efflorescence were sodium and potassium sulfates; and that cements with an alkali content over 0.6 per cent were consistently associated with mortar disintegration.
4. Cement was the only apparent source of appreciable amounts of sodium and potassium and the most consistent source of $SO_3$.

When combined, these items of evidence make it appear highly probable that the alkali content of the cement was the prime source of the sodium and potassium sulfates in the efflorescence, but other sources may have contributed $SO_3$.

Apparently, cements with an alkali content of 0.6 per cent or less did not produce sufficient destructive efflorescence to cause mortar disintegration in the majority of cases; but where the alkali content of the cement was more than 0.6 per cent, efflorescence on the mortar joints and mortar disintegration occurred consistently.

## Summary and Conclusions

The data in this paper were obtained from 64 brick buildings, subject to many variables in workmanship, materials, and construction practices. Some of the conclusions are necessarily tentative because of the limitations of the data:

1. Fifty-one of the sixty-four buildings studied had some degree of mortar disintegraton and 13 had none—a ratio of about four to one.
2. Eight of the nineteen buildings inspected in other areas showed mortar breakdown, and 11 had none.
3. The disintegration of mortar joints is progressive with time.
4. It generally took about two years after construction for mortar disintegration to appear.
5. Efflorescent salts forming at or near the surface of the mortar joints was the evident cause of the type of mortar disintegration studied.
6. Analysis of the efflorescent salts showed sodium and potassium sulfates to be the main components.
7. There was evidence that the alkali content of cement was the major source of the sodium and potassium in the sulfates in efflorescence which apparently caused the surface of the mortar joints to disintegrate.
8. The evidence, which however contained possible variables, indicated that

cements with more than 0.6 per cent of sodium and potassium oxides (soda equivalent) were consistently associated with mortar disintegration.

9. Apparently where cements with 0.6 per cent or less of sodium and potassium oxides (soda equivalent) were used, mortar disintegration either did not occur or, with one exception, was only slight.

10. Rough bonding surfaces on brick tended to decrease the mortar disintegration.

11. Brick with heavily sanded bonding surfaces tended to increase the mortar disintegration.

12. Good workmanship probably reduced the mortar disintegration, but its influence was not controlling.

13. Poor workmanship and high mortar joint cracking did not always cause mortar disintegration, but probably tended to increase it where there were destructive efflorescent salts in the mortar.

14. There was no satisfactory evidence that any significant influence was exerted on the mortar disintegration by the lime used, the sand used, double tooling of the mortar joints, or the temperature of the brick laying period.

15. Weather conditions favorable to the formation of efflorescence were described.

## *Acknowledgments:*

The authors wish to acknowledge their indebtedness to the cement manufacturers who furnished typical analyses of their products; to P. L. Rogers of the Riverton Lime and Stone Co., L. J. Minnick of G. & W. H. Corson, P. L. Bock of Warner Co., and T. F. Hart of Union Carbide and Carbon Co., who furnished material assistance and advice; and to T. Ritchie of the National Research Council of Canada for his research on efflorescence and his valued comments.

## REFERENCES

(1) T. Ritchie, "Study of Efflorescence on Experimental Brickwork Piers," *Journal*, Am. Ceramic Soc., Vol. 38, No. 10, p. 357 (1955).

(2) R. K. Robertson, "Efflorescence," *Journal*, Canada Ceramic Soc., Vol. 13, p. 40 (1944).

(3) C. C. Connor, "Some Effects of the Grading of Sand on Masonry Mortar," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 933 (1953).

(4) L. A. Palmer, "Wet Walls and Efflorescence," publication of the American Face Brick Assn., Am. Inst. Architects File No. 5m (1928).

(5) T. Ritchie, "Study of Efflorescence Produced on Ceramic Wicks by Masonry Mortars," *Journal*, Am. Ceramic Soc., Vol. 38, No. 10, p. 362 (1955).

(6) B. Butterworth, "Some Effects of Soluble Salts in Clay Products," *Transactions*, Ceramic Soc. (London), Vol. 36 (1937).

(7) P. L. Rogers, "Developing a Test Method for Efflorescence of Masonry Mortars," ASTM BULLETIN, No. 200, September, 1954, p. 64 (TP190).

# DISCUSSION

Mr. R. E. Copeland.[1]—Does the author have any evidence that the use of pozzolanic material in the mortar would reduce the efflorescence and the accompanying disintegration?

Mr. C. C. Connor (*author*).—No pozzolanic material was used in the mortars studied. I have no evidence regarding the possible effect of such a material.

Mr. C. T. Grimm.[2]—I would like to ask the author if colorless waterproofers were used on any of these buildings, and if there is any correlation between disintegration of the mortar joints and the use of this material.

Mr. Connor.—Colorless waterproofing was not used on any of the buildings in this study.

Mr. John J. White.[3]—The author spoke of the most severe disintegration of joints occurring on heavily sanded brick. If by that is meant glass block, glass brick, I should like an explanation of that, and if it is not meant to cover glass brick I wonder if there has been any experience on that type masonry unit.

Mr. Connor.—The indications were that clay brick having heavily sanded bonding surfaces increased the cracking between brick and mortar materially. This was true because the sand surfacing was loosely bonded in many cases. If much of the sand could be scraped off with a knife blade vigorously applied, the shrinkage of mortar would pull off the loose layer and a high degree of mortar joint cracking would occur. This cracking increased the amount of water entering the wall and the possibility of efflorescence. If heavily sanded brick were used with mortar containing a low-alkali cement, as at building No. 55, no mortar disintegration occurred; but if such brick were used with mortars containing much destructive efflorescent salts, the probability of mortar disintegration was increased.

Lightly sanded brick did not exert the same adverse effect as those that were heavily sanded, even though the sand was very loose. Loose sanding of any kind is not looked upon favorably, but with a light sanding, much of the loose sand is shed in laying and handling; and a good workable mortar will usually work through the remains of a light sanding and bond to the solid clay body underneath.

These statements do not apply to glass block. None was used at the buildings in the study, but, as a rule, these blocks do not have a loose layer of sand at the bonding surface.

Mr. J. W. McBurney[4] (*by letter*).—The authors have performed a great service in calling attention to this type of mortar disintegration and in having considered so many possible factors. That crystalization of soluble salts in the pores of masonry materials is a cause of disintegration was recognized by Brard[5] as early as 1828. Unfortunately, most of the research in the United States has been confined to salt action on heavy clay products and aggregates and, more particularly, to attempted correlations with freezing-and-thawing tests. Butterworth's chapter on the "Properties of Clay Building Materials"[6] presents a critical review of the literature on efflorescence and salt action on heavy clay products up to and through 1950. A very recent paper by Watson[7] presents evidence on the increased damage to brick by freezing and thawing when the pores near or at the surface are choked by deposition of salts. It is my conviction that visible efflorescence is not the cause of disintegration but rather it is what happens below the surface that causes trouble. This is the "cryptoflorescence" of Cooling.[8] Furthermore, the distribution of visible efflorescence between the brick surface and the exposed mortar joints does not necessarily indicate the original source of the soluble salts. The principal factor is the relative permeability of the brick and the mortar when water and soluble salts are in the masonry. That the observations reported by the authors are explained by salt action is accepted. The rest of this discussion is on sources of these salts.

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[2] Assistant Director, Engineering and Technical Structural Clay Products Inst., Washington, D. C.

[3] Chief Specifications Engineer, Parsons, Brinckerhoff, Hall, & Macdonald, New York, N. Y.

[4] Consultant, Silver Spring, Md.

The authors apparently consider that the soluble salts must have been in the masonry units or mortar ingredients at the time of construction. They mention $SO_3$ in the air of industrial areas but refer to "the virtual elimination of other possible sources of the alkali sulfates found in the efflorescence" with the conclusion that portland cement is the most probable source of alkali sulfates. The values for $SO_3$ given in Table X probably represent gypsum added to control set. There is general agreement that during setting of portland cement, gypsum unites with calcium aluminate to form insoluble calcium sulfoaluminate. Whether this compound will decompose and, if so, under what conditions, is apparently controversial. Addition of soluble sulfate to set and hardened mortar is a well-known cause of expansion and disintegration. It is suggested that sulfate from the atmosphere rather than from the portland cement is the source of much of the $SO_3$ in the authors' one analysis of efflorescence (Table IV).

Searles-Wood[9] calculated that 16 million tons of coal would add enough sulfur dioxide gas to the atmosphere to convert half a million tons of limestone into 860,000 tons of gypsum. A recent paper by Greenberg and Jacobs[10] reported that the 30 million tons of coal and fuel oil equivalent, burned annually in greater New York supplied one million tons of $SO_2$ in the air, which converted to over two million tons of sulfuric acid ($H_2SO_4$). The concentration of $SO_2$ in the New York air approached 0.80 ppm in December and January and fell to 0.3 ppm in July and August.

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[5] Brard, *Annales de Chimie Physique*, Vol. 38, p. 160 (1828).

[6] "Ceramics, A Symposium," *Transactions*, British Ceramic Soc., pp. 824–877 (1953).

[7] A. Watson, "Surface Growth of Salt Crystals as a Contributing Cause of Frost Damage to Bricks," *Transactions*, British Ceramic Soc., Vol. 56, No. 7, July 1957, pp. 366–368.

[8] L. F. Cooling, "Contributions to the Study of Florescence, II.-Evaporation of Water from Brick," *Transactions*, British Ceramic Soc., Vol. 29, No. 2, pp. 39–52 (1930).

[9] H. D. Searles-Wood, "The Deterioration of Buildings," *Power Engineering* (London), Vol. 24, No. 280, p. 206 (1929).

[10] L. Greenberg and M. B. Jacobs, "Sulfur Dioxide in New York City Atmosphere," *Industrial and Engineering Chemistry*, Vol. 48, No. 9, pp. 1517–1521 (1956).

The extensive sulfating of Indiana Limestone exposed in the atmosphere of Cleveland, Ohio, for less than 20 yr was personally observed in 1923. One of the worst examples of sulfate attack was located on the campus of Western Reserve University.

Three items in the authors' paper which are (1) the date 1944, (2) that the $Na_2O$ content of their one analysis of efflorescence exceeded the $K_2O$ content by a ratio of approximately 3 to 2, and (3) that the one locality with 100 per cent incidence of disintegration was Long Island, lead to the suggestion that consideration be given to the activities of certain "ladies" known as Carol, Edna, Hazel, Connie, and Diane plus an anonymous pair who visited the region in 1938 and 1944 respectively.

A hurricane by definition is a wind having a velocity of 75 mph or greater. The Weather Bureau reported 186 mph at the Blue Hill Observatory of Harvard University, Mass. in 1938. The Weather Bureau's description of the effect of a hurricane on a body of water is "Air filled with foam and spray. Sea completely white with driving spray. Visibility very seriously affected." North Atlantic hurricanes rotate counterclockwise with the highest velocities in their north-east quadrant. Very heavy rains are associated. For example, Connie (1955) gave 12.20 in. of rain at LaGuardia Field, New York. The mineral composition of sea water is well known. The authors' statement that sodium and potassium in the air "are hardly possible" is accordingly challenged. The data presented by Egner and Erikson[11] are recommended for consideration. What follows is a brief summary of their data. These authors reported monthly totals of the mineral content of air and rain collected at a large number of stations in Sweden along with corresponding data from Edinburgh and Aberdeen, Scotland and Leeds and Rothampstead, England. The values for sodium and potassium are for the elements and are expressed as milligrams per cubic meter. The maximum value for sodium was reported for February 1955 at the Vinga Station. This was 4870 mg per sq m with an associated value of 180 for potassium. High values were reported from Liska with a maximum of 3880 for sodium. Liska usually exceeded 1600 mg per sq m for the sodium during the fall and winter months. Both Vinga and Liska are on the coast. Kiruna, inland in north central Sweden, has much lower values for sodium and potassium. The maximum was 33 mg for sodium in rain with 1 mg for potassium reported at the same time (November 1955). The majority of the Kiruna values did not exceed 5 mg per sq m for sodium. The maximum sodium content for air (not rain) was 156 kg per cu km reported from Liska for July 1956. The corresponding potassium was 6.9 kg. The same month gave 470 mg per sq m for sodium and 23 for potassium in rain at the same station. It should be pointed out that Sweden does not have hurricanes. Their winter storms would be "gales" (35 to 54 mph) or at the most "whole gales" (55 to 74 mph). That there is salt in the atmosphere can be personally vouched for by the taste of the overnight deposits on the windshield of an automobile parked about half a mile from the Pacific in the neighborhood of Los Angeles during August 1950. During the time of observation the wind was light (1 to 7) or at the most gentle (8 to 12 mph).

Diane, which grazed Washington,

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[11] H. Egner and E. Erikson, "Current Data on the Chemical Composition of Air and Precipitation," *Tellus* (Stockholm), Vol. 7, No. 1, pp. 134–139; No. 2, pp. 261–271; No. 3, pp. 395–400; No. 4, pp. 522–529 (1955); Vol. 8, No. 1, pp. 112–113; No. 2, pp. 283–284; No. 3, pp. 411–412; No. 4, pp. 513–516 (1956).

D. C., in August 1955, was below hurricane rating with respect to wind velocity after coming ashore. Her principal contribution was rain (8.72 in. in 3 days with a maximum day's average speed of 24.7 and highest speed of 49 mph at air port). Her aftermath was efflorescence in quantities and locations previously unobserved. This was dismissed at the time as the expected result of a prolonged heavy rain accompanied by high winds. It was negligence that the masonry exposure test plot at the Nat. Bureau of Standards was not inspected subsequently. Soluble salts had apparently been completely leached out of the unprotected wallettes some years before. The final inspection in January 1956 showed nothing except the insoluble calcium carbonate scumming noted in the 1956 paper.[12]

The item of the ratio of sodium to potassium salts warrants some further mention. Of 15 analyses made on Lehigh Valley portland cements, the $K_2O$ exceeded the $Na_2O$ in all cases. The minimum difference was 0.53 $K_2O$ to 0.40 $Na_2O$ in per cent. Eleven of the analyses gave the $K_2O$ content as at least twice that of the $Na_2O$. More $K_2O$ than $Na_2O$ was found in all the 5 specimens of the Hudson Valley product. The question has been asked what becomes of the chloride if any considerable percentage of the sodium and potassium comes from sea water. A probable explanation of the disappearance of the chlorine is that it is replaced by sulfate and disappears as hydrogen chloride (HCl).

In conclusion, it is not denied that portland cement is a source of soluble alkali. It is suggested, however, that for the localities cited by the authors seaborne alkalies are probably an important factor. Potassium and sodium salts with their relatively high solubility will usually leach out of masonry in a comparatively short time. Additional salts from the air, as suggested, would appear to be a necessary requirement for continued damage. The authors are again thanked for their contribution.

MR CONNOR.—Mr. McBurney has very properly pointed out the possibility that sodium and potassium salts in the atmosphere at the seashore, and inland for some distance after hurricanes, might have contributed to the formation of the alkali sulfates which caused the mortar disintegration described in the paper. He evidently thinks that the sodium and potassium chlorides could in some way combine with $SO_3$ from some source and form sulfates. Mr. McBurney challenged the statement in the paper that sodium and potassium in the air are hardly possible. That statement was made in connection with the discussion of $SO_3$ in industrial atmospheres, and was essentially correct. However, it must be admitted that sodium and potassium salts could have been in those atmospheres during hurricanes, and I thank Mr. McBurney for pointing it out.

The possibility of sodium and potassium salts in the air was considered but not presented in the paper. The following items of evidence indicate that airborne sodium and potassium salts did not contribute consistently nor materially to the alkali sulfates that caused the disintegration.

1. The 1938 hurricane was most severe and penetrated deep into the area covered by the study, hitting many of the same company's buildings. No evidence of the type of mortar disintegration described was ever observed at any of these buildings. The same statement applies to the many buildings in the area covered by the 1944 hurricane.

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[12] J. W. McBurney, "The Effect of Weathering on Certain Mortars Exposed in Brick Masonry With and Without Caps and Flashings," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 1283 (1956).

2. Buildings Nos. 53, 54, 55, 62, and 63 were buildings *without* mortar disintegration which were near the ocean or bays. Building No. 53 in Atlantic City was only one block from the ocean and had been constantly subjected to airborne sea salts since 1947. All these buildings were hit hard by several of the hurricanes and should have shown severe mortar disintegration if the airborne sodium and potassium salts had contributed materially to the alkali sulfates. None did.

3. The 64 buildings in the study showed no evident tendency for the severity of the mortar disintegration to increase at buildings relatively near the salt water.

4. All of the buildings *without* mortar disintegration were hit by some of the hurricanes mentioned by Mr. McBurney, but the airborne sea salts had no effect.

5. Most of the series of 43 efflorescence-free buildings built in the seven year period preceding this study were battered by several of the hurricanes. They were not examined immediately thereafter, but subsequent observations showed no efflorescence and no mortar disintegration. Moreover, buildings Nos. 15, 27, 32, and 47 which *did* show efflorescence and mortar disintegration were extensions or additions to four of these efflorescence-free buildings.

The comparison of building No. 15, a 1952 extension, with its original section built in 1941 furnished most convincing evidence that some difference in the mortar materials was the probable cause of mortar disintegration rather than airborne salts. Eight miles from the ocean, the two sections had both been subjected to the same storms and airborne sea salts. Both sections had the same brick and the same high-lime mortar mix, and were almost exact duplicates in design and construction. However, the brands of cement and lime and the kind of sand were not known, but the sands were undoubtedly from nearby inland sand pits. The original 1941 section was entirely free from efflorescence and mortar disintegration, but No. 15, the 1952 extension, showed considerable efflorescence even in a somewhat unfavorable period, and the mortar disintegration was severe.

6. Mr. McBurney laid considerable stress on the fact that the buildings in the study were located near the ocean. Ritchie[13] found a predominance of sodium and potassium sulfates in the efflorescence on his brick piers in Ottawa, Canada. Those piers were some 300 miles from the ocean and out of the usual path of hurricanes. Ritchie found also that where efflorescence occurred, the amount of efflorescence generally increased with the proportion of the cement in the mortars.

The foregoing items furnish strong evidence that airborne sea salts had no material influence on the mortar disintegration studied.

Mr. McBurney suggests that sulfate from the air rather than from the cement was the source of much of the $SO_3$ in the efflorescence. It was stated in the paper that sources other than cement may have contributed $SO_3$. Nevertheless, such contributions could not have been an important factor. Nine of the 13 buildings without mortar disintegration were in heavily industrialized areas and were also in areas covered by some of the hurricanes. According to Mr. McBurney's thesis, these buildings should have developed mortar disintegration. But they did not. Even the combination of airborne sea salts and considerable $SO_3$ in the air did not contribute sufficiently to the alkali sulfates to cause mortar disintegration at these buildings.

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[13] T. Ritchie, "Study of Efflorescence on Experimental Brickwork Piers," *Journal*, Am. Ceramic Soc., Vol. 38, No. 10, p. 357 (1955).

There was no evidence in the data that mortar disintegrations was increasingly severe in highly industrialized areas where concentrations of $SO_3$ in the air could be expected.

Mr. McBurney pointed out that the amount of sodium sulfate found in the destructive efflorescence exceeded that of the potassium sulfate which reversed the amounts of sodium and potassium oxides in the cements he had analyzed. I have no explanation. Ritchie[14] also found the sodium sulfate greater in his analyses of efflorescence from brick piers. Furthermore, Ritchie[14] found that the sodium carbonate in efflorescence on ceramic wicks in one week old mortar also exceeded the potassium carbonate which reversed the amounts of sodium and potassium oxides found in the cements used.

I would also like an explanation for the apparent fact that the early efflorescence on mortar in brickwork is predominantly sodium and potassium carbonates, and that large amounts of the alkali sulfates do not usually form until a year or more has elapsed. The two explanations might be related.

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[14] T. Ritchie, "Study of Efflorescence Produced on Ceramic Wicks by Masonry Mortars," *Journal*, Am. Ceramic Soc., Vol. 38, No. 10, p. 362 (1955).

# THE PERMEABILITY OF SOILS AND THE CONCEPT OF A STATIONARY BOUNDARY-LAYER*

BY WERNER E. SCHMID[1]

## SYNOPSIS

The complex problem of determining the influence of different variables on the permeability of soils is investigated by establishing a working model which allows the derivation of a theoretical expression for the permeability.

Results of tests conducted by the author and several other investigators are used to show the general validity of the derived expression. A discussion follows giving theoretical as well as experimental evidence why some at present still widely used concepts regarding the relationship between void ratio and permeability can not be valid and the limits of validity of the proposed expressions are investigated.

The problem of permeation of a fluid through a medium of granular solids is an intricate and complex one. Since Darcy in 1856 discovered the fundamental law: $Q = v \cdot A = k \cdot \frac{H}{L}$, that is, the discharge $Q$ through a porous medium of area $A$ is directly proportional by a factor, $k$, to the first power of the hydraulic gradient $\frac{H}{L}$, scientists and engineers from many nations have tried to investigate the variables which influence this proportionality factor, $k$, or, as it is frequently called, the coefficient of permeability.

Particularly in the past 50 years, because of the growing importance of the problem in our modern technology, numerous attempts have been made to attack the problem theoretically as well as experimentally; and the existing literature on the subject is tremendous.

However, if we expect a universally valid relationship as a result of this extensive research effort—even within reasonably practical limits—we are being disappointed. Alone for the influence of the void ratio or the porosity of the granular medium we discover at least ten competing and conflicting expressions (Franzini, **12**),[2] many of them supported by or derived from experimental results.

One explanation for these various discrepancies and contradictory results is that many of the earlier investigations had a rather limited purpose. Often an

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Assistant Professor, Department of Civil Engineering, Princeton University, Princeton, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1210.

answer was required for a rather specific problem, for example, the permeability of a rather uniform filter sand or of a certain oil-bearing formation. Hence, some of the proposed expressions were "tailored" to fit particular test conditions. Also, experimental data over a wide range of porosities for the same medium are rather scant and fairly recent. Most earlier experiments were conducted on uniform sands, lead shot, glass beads, or steel balls of equal diameter, and with various fluids such as air, water, oils, etc. In an excellent and very fundamental treatise on the packing of spheres, Graton and Fraser (**15**) showed that the porosity, $n$, of such media can vary theoretically only between $n = 0.2595$ and $n = 0.4764$ and, for all practical purposes, will be somewhere between 0.30 and 0.40. That is to say, the void ratio will vary between 0.42 and 0.67. Thus, most results obtained ran over a very limited range of porosities—too limited in our opinion to permit extrapolation over a wider range. In summary it may be stated that none of the various proposed equations has been tested severely over a wide range of conditions or where this has been done the results were inconclusive.

A second explanation for the unsatisfactory correlation of the numerous earlier test results is the suggestion that at least one of the important variables entering into the permeation problem has not been discovered or was disregarded in the evaluation of such results. If such is the case, we can immediately deduct that those data which do not include all the controlling factors are not comparable. Another point supporting the above suggestion is the fact that, despite all efforts, we have so far been unable to correlate test results with the theoretical equation which should be most closely related to the permeation problem: that is, the Hagen-Poiseulle equation for laminar flow through a capillary tube. In general all fluid flow equations (Blasius, Nikuradse, Weissbach, Chezy, Manning, Hazen-Williams) are actually empirical. That is to say, they are either inducted from test results or, although deducted from some fundamental assumptions, are nevertheless fitted to experimental data by empirical coefficients.

Hence, it would seem logical to use the Hagen-Poiseulle equation and determine the correlating coefficients from experimental data. Such a procedure was previously used rather successfully by Winterkorn (**32**) in the investigation of the slaking behavior of dry soils.

## STATEMENT OF PROBLEM

Approaching the subject from a most general point of view, it will be advantageous to restate the problem: We will investigate the factors which influence the movement of a fluid through a porous medium under gravitational or body forces. Such movement shall be called permeation.

Let us call the moving fluid the permeant, the medium being permeated we shall call the permeate, and the two together we will call the permeation system. Let us also confine our considerations to the case of steady and laminar flow and accept the validity of Darcy's law. In order to eliminate all the factors not readily accessible to a quantitative analysis, we will make the further restriction of assuming that the permeation system is stable. A stable system shall be one in which none of the following phenomena occur or are negligible during permeation:

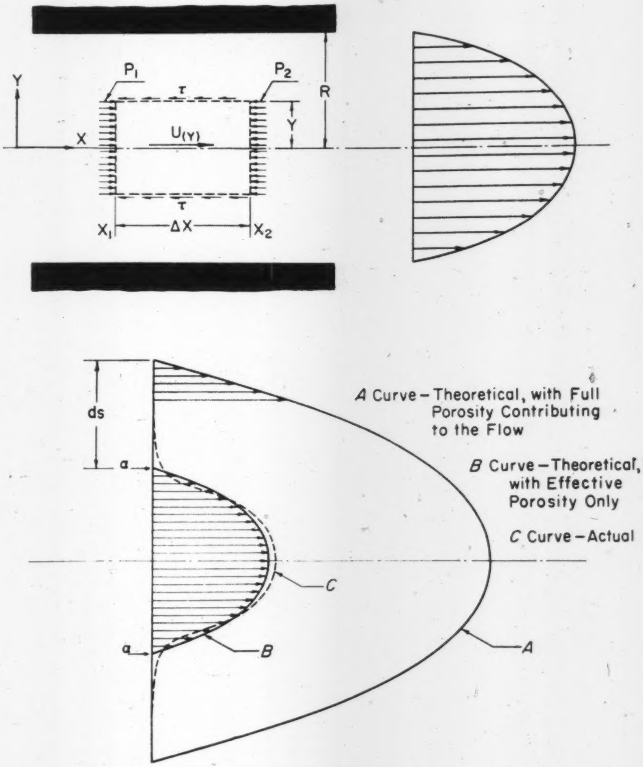
1. Volume changes of permeate or permeant;

2. Ion exchanges, or dissolution and leaching of the permeate;

3. Deposition of solid, liquid, or gaseous matter by the permeant within the

permeate (air bubbles, etc.);

4. Structural changes of the permeate due to dispersion, aggregation, or changes in the crystal lattice; and
5. Temperature changes.


P₁
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A Curve – Theoretical, with Full Porosity Contributing to the Flow
B Curve – Theoretical, with Effective Porosity Only
C Curve – Actual
C
a
B
A

FIG. 1.—Longitudinal Section through Ideal Flow Channel and Velocity Distribution.
(a) Flow in a circular cylindrical tube.
(b) Velocity profiles.

In addition, we assume that the statistical averages for the number, size, and distribution of the pores in the permeate is constant for all cross-sections normal to the direction of flow—that is, we assume the permeate to be macroscopically homogeneous.

For most practical cases the above restrictions will not limit the applicability of our considerations. In general a stable system will be achieved some time after permeation begins. Otherwise we are dealing with a physical or chemical process of no interest to our investigations.

It will be obvious that the factors affecting permeation will be determined by the properties of permeate and permeant. However, it seems illogical to describe the permeability factor, *k*, by a juxtaposition or separation of the individual material properties of permeate and permeant, since the problem is one of mutual composite interaction. Just as we cannot describe the interaction of a composite steel and concrete beam by moment of inertia and modulus of elasticity of steel and concrete alone but rather have to find their effective moment of inertia about the composite neutral axis, we should not expect to be able to describe fully the permeability of a permeation system by some more or less arbitrary separation of the individual material properties. The term permeability factor (or permeability) rather than coefficient of permeability was deliberately used above for *k*, since as we will show it is neither a dimensionless coefficient nor a material constant but rather a property of the permeate-permeant system. Accepting the premise of the interaction between solid and fluid, let us then investigate its nature and extent. Before that, however, it will be necessary to make some general considerations.

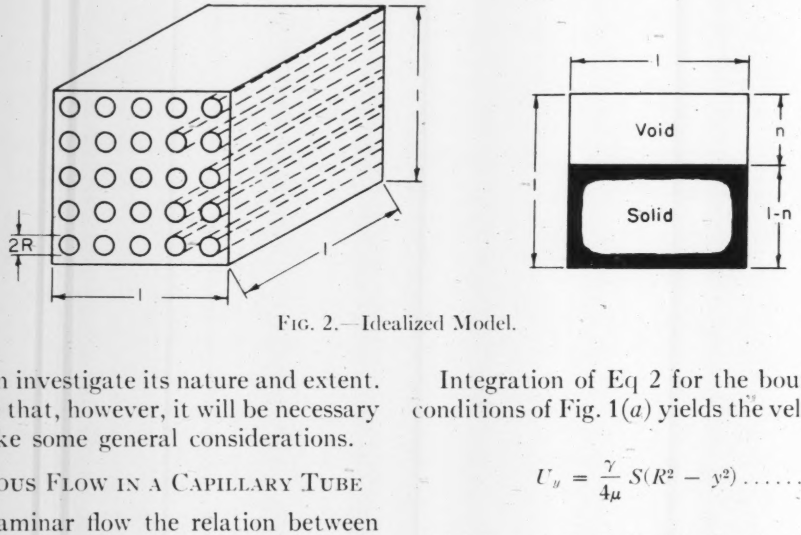


FIG. 2.—Idealized Model.

## VISCOUS FLOW IN A CAPILLARY TUBE

In laminar flow the relation between the shearing stress $\tau$ and the velocity $U$ for adjacent laminae is expressed by the differential equation:

$$\frac{dU}{dy} = -\frac{\tau}{\mu} \quad \ldots\ldots\ldots\ldots (1)$$

where $\mu$ is the absolute viscosity.

For a circular cylindrical tube (Fig. 1(*a*)), the average shear stress between two cross-sections $x_1$ and $x_2$ may be found as:

$$\tau_A = \frac{\text{force}}{\text{area}} = \frac{y^2\pi(p_1 - p_2)}{2y\pi(x_2 - x_1)} = \frac{y}{2}\cdot\frac{\Delta p}{\Delta x}$$

and using the unit weight of the liquid

$$p = \gamma \cdot h: \quad \tau_A = \frac{y\gamma\Delta h}{2\,\Delta x}.$$

In the limit $\Delta x \rightarrow 0$ the average shear stress approaches the true value:

$$\tau = \lim_{\Delta x \to 0} \tau_A = \frac{y\gamma}{2}\frac{dh}{dx} = \frac{y}{2}\gamma S$$

$$\left(\frac{dh}{dx} = S = \text{hydraulic gradient}\right).$$

Hence Eq 1 reads:

$$dU = -\frac{y\,dy}{2\mu}\gamma S \quad \ldots\ldots\ldots (2)$$

Integration of Eq 2 for the boundary conditions of Fig. 1(*a*) yields the velocity:

$$U_y = \frac{\gamma}{4\mu} S(R^2 - y^2) \quad \ldots\ldots\ldots (3)$$

which has a parabolic distribution across the section. As the volume of a paraboloid of revolution is $\frac{1}{2}$ area times height, the mean velocity $U_m$ through the tube can be written:

$$U_m = \frac{\gamma}{8\mu} R^2 S \quad \ldots\ldots\ldots (3(a))$$

This equation first deduced by Hagen (**16**) and shortly afterward by Poiseulle (**25**) is the well known and previously mentioned Hagen-Poiseulle equation for laminar flow through a tube.

### RELATIONSHIP BETWEEN THE HAGEN-POISEULLE AND THE DARCY EQUATION

We write Darcy's law in its original form as:

$$Q = v \cdot A = k \cdot S \cdot A, \ldots\ldots\ldots (4)$$

where $Q$ is the discharge (cu cm per sec), $v$ the filter velocity (cm per sec), $A$ the total area of the soil (sq cm) subjected to the hydraulic gradient $S$, and $k$ (cm per sec) the permeability factor.

If we want to correlate Eqs 3(*a*) and 4, we have to convert the mean velocity $U_m$ in a capillary tube into the filter velocity $v$ over the total soil cross-section. For this purpose we picture the pores in a unit cube of soil concentrated to form a series of uniform circular tubes of radius $R$ having a length of unity (Fig. 2). Then, using Eq 3(*a*), the discharge through each of these tubes will be:

$$Q_T = U_m \cdot \pi \cdot R^2 = \frac{\pi\gamma}{8\mu} \cdot R^4 \cdot S$$

If there are $m$ such tubes in a unit cross-section the total discharge per unit area will be:

$$Q = m \frac{\pi\gamma}{8\mu} R^4 \cdot S$$

As the porosity $n$ of our idealized model is: $n = m\pi R^2$, we may substitute it in the above equation as:

$$Q = \frac{\gamma}{8\mu} R^2 \cdot n \cdot S = \frac{\gamma}{32\mu} D^2 \cdot n \cdot S \ldots . (5)$$

Since Eq 5 now gives the discharge through a unit cross-section, we may equate Eqs 4 and 5:

$$v = kS = \frac{\gamma}{32\mu} D^2 \cdot n \cdot S$$

and hence

$$k = \frac{D^2 \gamma n}{32\mu} = CD^2 \frac{\gamma}{\mu} n \ldots\ldots\ldots (6)$$

The first attempt to correlate the filter velocity $v$ and the average interstitial velocity $U_m$ is attributed to Dupuit (**8**).

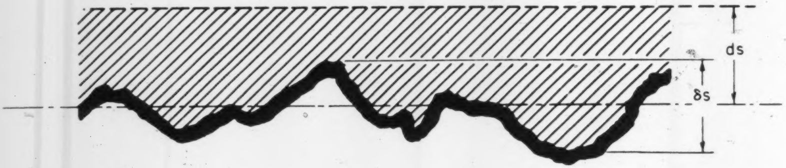
According to Eq 6 the permeability, $k$, should be directly proportional to the porosity, $n$, and to the square of some effective diameter, $D$. To our knowledge nobody has ever succeeded in proving the validity of this formula by experimental observation.

### THEORY OF A STATIONARY BOUNDARY-LAYER

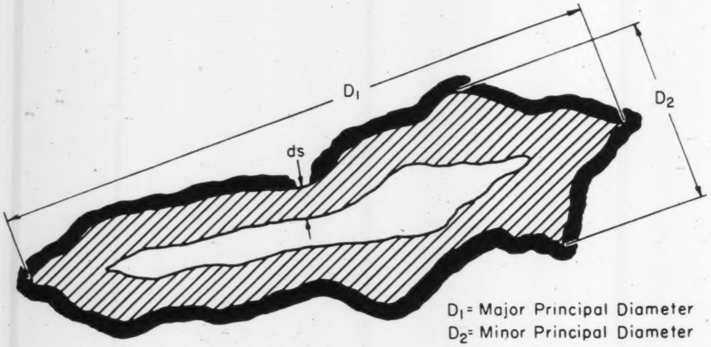
For the purpose of mathematical analysis, the modern theory of fluid dynamics conveniently divides the field of flow into two regions. In the first region the forces due to friction are small and negligible and the theory of perfect fluids offers very good results. The second region, where friction must be taken into account, is the thin so-called boundary-layer at the fluid-solid interface (Prandtl, **26**). This separation brings about a considerable simplification of the mathematical analysis. In fact, theoretical studies were made possible only after the introduction of this boundary-layer concept.

In our problem, throughout the permeation system we deal with viscous laminar flow only, that is with the classical boundary layer flow alone. It may seem farfetched to elaborate on these well known hydrodynamic principles, yet there seems to be a striking analogy.

Let us return to the problem of interaction between the permeate and permeant. If there is such interaction, it must occur at the fluid-solid interface. The smoothest physical surface is the cleavage plane of a single crystal. Estermann and Stern (**10**) showed, by observing the reflection of a beam of molecular rays of helium on the cleavage plane of a LiF crystal, that the order of magnitude of the irregularities of such a surface is of the order $1 \times 10^{-8}$ cm and is due to the thermal movement of the ions. In comparison with such crystal cleavage planes, ordinary so-called smooth surfaces are actually extremely rough and have protuberances which are of several hundred molecular diameters and are of the order of magnitude of $10^{-5}$ cm. In the case of normal granular media the height $\delta$ of the protuberances may be many times this distance and probably is of the order of $\delta = 10^{-4}$ cm or 1 $\mu$. Thus we should consider the fluid-solid interface not as a plane but as an undulating surface with peaks, valleys and craters as shown in Fig. 3(*a*). Quite obvious fluid particles in the valleys will be almost stationary and will contribute little to the flow. Still more important, however, is the fact that along the surface of the solid chemical or electrical surface forces do exist (heat of wetting) which may be of different origin. First there are the so-called broken bond forces caused by the interruption at the surface of the normal sequence and balance of the molecular or atomic force fields within the crystal lattice. The surface atoms thus may tend to establish bonds with adsorbed atoms by sharing electrons or orbitals. Another type of surface energy are the Van der Waal's forces which cause bonding of adsorbed molecules by lowering of the total energy of the system through mutual harmonic motion of the electrons in the electron clouds.



(*a*) Typical surface profile.



(*b*) Cross-section through typical pore channel.

FIG. 3.—Pore Surface and Pore Section.

The mechanism of water fixation (hydration) at the surfaces of soil particles thus may be of a twofold nature: one would be an orientation of the fluid molecule at the surface due to the electrical properties of both the liquid and the surface. This type of hydration is accompanied by the release of energy (heat of wetting) and Winterkorn calls it hydratation (**33**). In the second the osmotic type of hydration hydratated ions surround the hydratated surface in a diffuse layer (**34**). These ions keep within the ionic atmosphere of the surface. This ionic atmosphere is the distance within which the mean osmotic force is in equilibrium with the mean electrical attraction due to the different charges of the surface and the exchangeable ions. The free liquid in which the hydratated ions are dispersed is an integral part of the fixed layer. Since the diffuseness of the double layer is a function of the osmotic pressure of the cations this type of fixation may be termed osmotic (**33, 34**).

Thus, we should expect a stationary film of fluid particles along the surface of the top protuberances of the solid (Fig. 3). Its thickness $d_s$ will vary with the surface energy of the solid and the electrical properties (dipole moment and dielectric constant) of the fluid. For the purpose of our investigation we may assume $d_s$ = constant for any given permeate-permeant system.

The previously mentioned analogy now becomes obvious: in ordinary hydrodynamic theory we separate the field of flow into two regions, one for which the perfect fluid theory approximately holds, the second where the laws of viscous flow hold. In laminar boundary-layer flow we separate the flow field into a region where the theory of viscous fluid flow governs and one—the stationary boundary-layer—where it is no longer applicable because this region is for all practical purposes without movement. A whole series of observations of the ordinary boundary-layer theory (**27**) now may become applicable either immediately or after some analogous interpretation. Here we think in particular of the problem of boundary-layer stability, separation, the so-called wake behind blunt edges, and the increase in boundary-layer thickness in the downstream direction. But all this would lead us too far away from our immediate problem. We would like to state, however, that the concept is perfectly general and applicable wherever it is necessary to investigate the viscous boundary-layer on a microscopic scale.

## THE CONCEPT OF THE STATIONARY BOUNDARY-LAYER APPLIED TO THE PERMEATION PROBLEM

Returning to our permeation system we will observe that the interstitial channels formed by the conjugate voids have in general a varying cross-section of some oblong shape as shown in Fig. 3(*b*). If we call $D_1$ the major principal diameter and $D_2$ the minor principal diameter, both of which may vary in magnitude and direction along the axis of the channel, we note that there can be no flow through this channel if the minor principal diameter $D_2$ is equal to or less than twice the stationary boundary-layer thickness $d_s$ at any point along the channel. The channel could be compared to a pipe with a closed valve. It is apparent that in a fine grained medium there will be many places where this condition $D_2 \leq 2d_s$ exists. Such a permeate will therefore have a large number of "pipes with a closed valve" and hence a low permeability. This, at last, provides a logically satisfactory explanation of why the fine grained soils of very high porosities exhibit such low permeabilities. We thus notice that only a small percentage of the total porosity contributes to the flow, the larger part being ineffective because of the presence of a "closed valve" at some point along the axis of most interstitial ducts and because of the adsorbed layers on the solid surfaces. When we determine the porosity of a granular fine grained medium, we usually determine the saturated unit weight and the dry unit weight; and, using the specific gravities of fluid and solid, we compute the void ratio and porosity.

For complete saturation the relationship between wet and dry density is given by $\gamma\text{dry} = \dfrac{\gamma\text{wet}}{1 + M}$ where $M$ is the fluid content for complete saturation in per cent of the dry weight.

$M$ is usually found by evaporation of the permeant under standard conditions. In the case of a water-soil permeation system it is normally done at 110 C for at least 4 to 6 hr. This procedure naturally causes evaporation of the fluid not only from the effective but also from the ineffective "locked up" channels as well as from the surface films formed by the hydrated ions. Reduction of this film proceeds until equilibrium is reached between the vapor pressure of the fluid molecules in the hydrated ions and the electrostatic force field of the ion. The porosity thus determined is the "total" porosity and will be very close to the "true" porosity. In order to get a measure of the effective porosity only, we will have to rely upon an indirect determination, since it is impossible to separate the permeant volume into its components, namely, the part that contributes to the flow, the part that is locked up in the ducts with a closed valve, and the amount which is actually adsorbed in the stationary boundary layer. Let us therefore modify our idealized working model in the following way (Fig. 4): We distribute the ineffective part of the porosity as hollow cylinders of outside radius $R$ and inside radius $R_E$ along the walls of the tubes of our original model. Then the total porosity $n$ will be, as before:

$$n = m\pi R^2 \quad \text{..........} \quad (7(a))$$

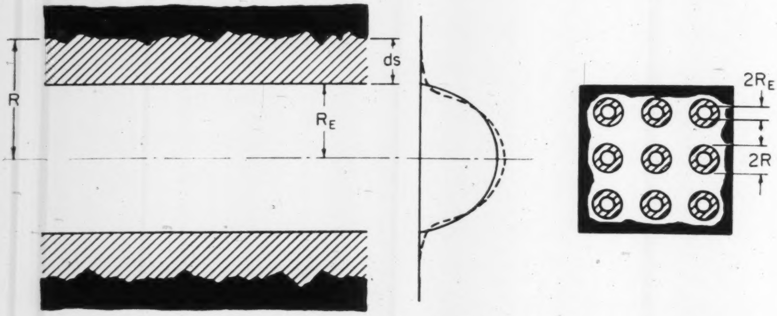


FIG. 4.—Modified Idealized Models.

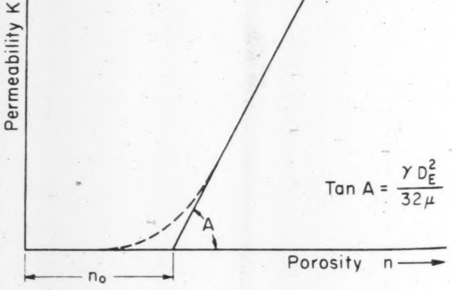


FIG. 5.—Porosity *versus* Permeability After Equation 8.

the effective porosity $n_E$ will be:

$$n_E = m\pi R^2_E \ldots\ldots\ldots (7(b))$$

and the ineffective porosity $n_0$ :

$$n_0 = n - n_E = m\pi(R^2 - R^2_E) \ldots (7(c))$$

Returning to Fig. 1(*b*), the theoretical velocity distribution which in the original model was described by curve *A* must now follow a parabola given by curve *B*. The true velocity profile is probably distributed as is shown in curve *C*, but the idealized profile *B* will be a very good approximation. If we now again apply the Hagen-Poiseulle equation to our revised model we get:

$$k = D^2_E \frac{\gamma}{32\mu} n_E = \frac{\gamma}{32\mu} D^2_E \cdot (n - n_0) \ldots (8)$$

where:

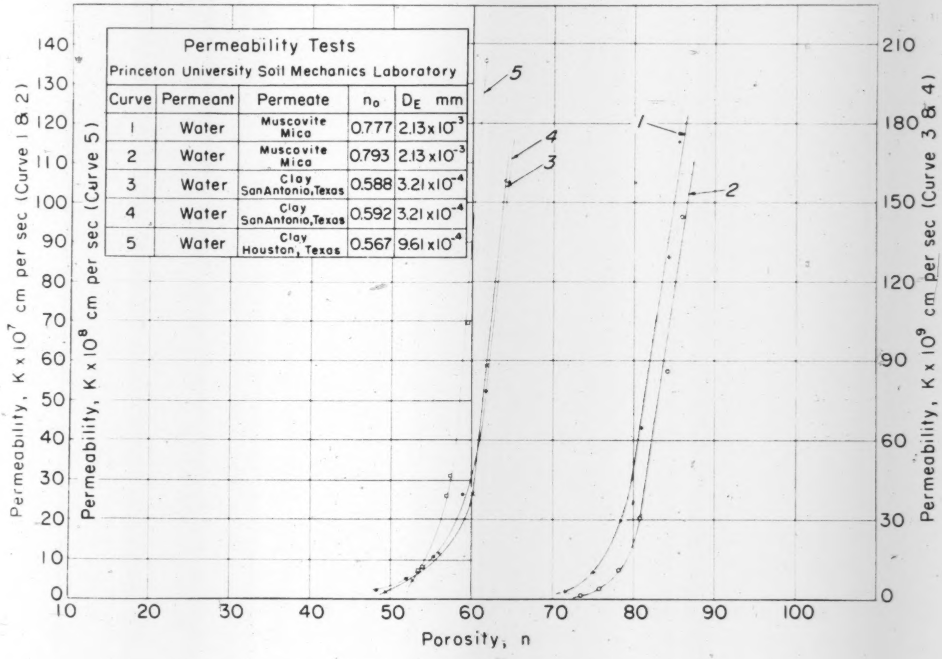
$D_E$ is the effective diameter equal to $2\ R_E$ ,
$n$ the total porosity,
$n_0$ the ineffective porosity, and
$\gamma$ and $\mu$ are the unit weight and the absolute viscosity of the permeant respectively.

In order to keep $k$ positive (as a negative $k$ has no physical significance) we require: $n - n_0 > 0$.

We shall now make two assumptions:

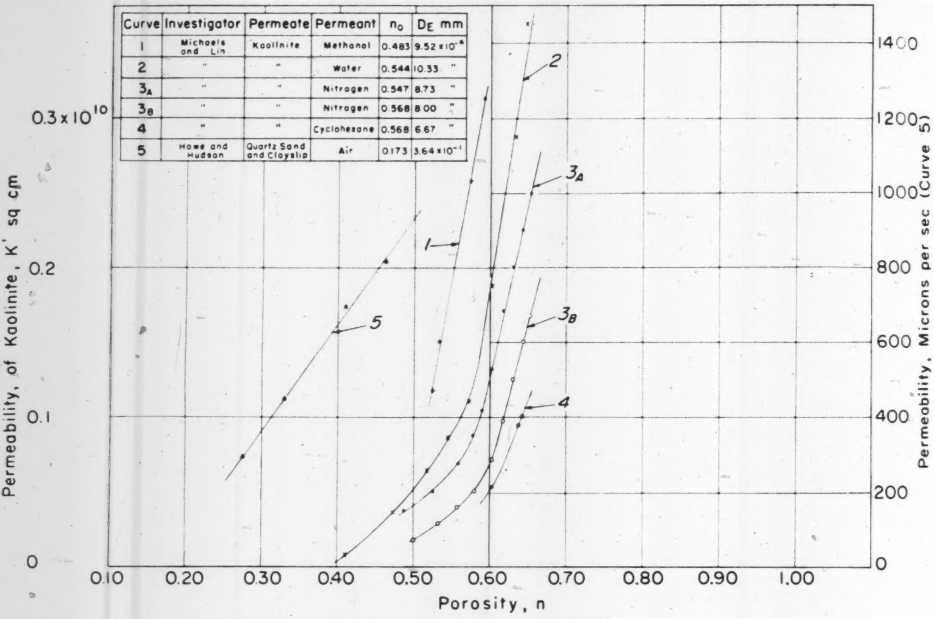
1. The effective diameter $D_E$ is primarily controlled by the granulometry of the permeate and is a constant for any given permeation system.

2. For any given permeate-permeant system at a given dispersion the ineffective porosity, $n_0$ is constant and



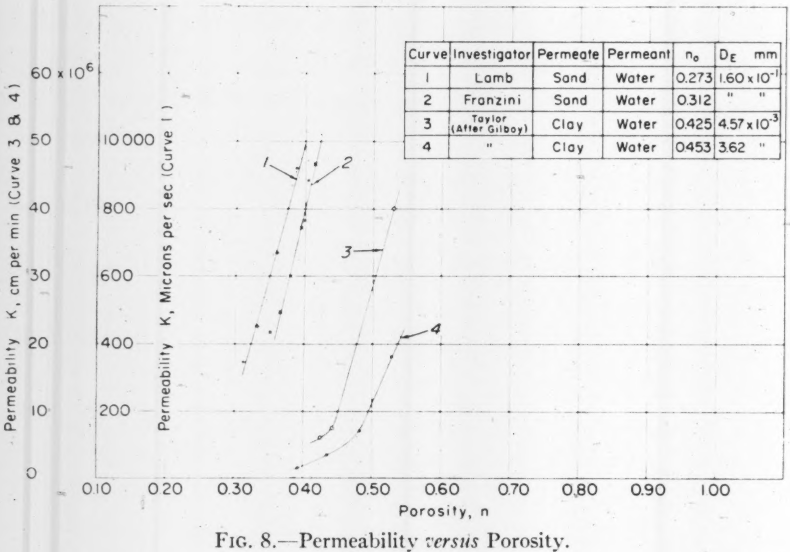
| Curve | Permeant | Permeate | $n_0$ | $D_E$ mm |
|---|---|---|---|---|
| 1 | Water | Muscovite Mica | 0.777 | $2.13 \times 10^{-3}$ |
| 2 | Water | Muscovite Mica | 0.793 | $2.13 \times 10^{-3}$ |
| 3 | Water | Clay San Antonio, Texas | 0.588 | $3.21 \times 10^{-4}$ |
| 4 | Water | Clay San Antonio, Texas | 0.592 | $3.21 \times 10^{-4}$ |
| 5 | Water | Clay Houston, Texas | 0.567 | $9.61 \times 10^{-4}$ |

FIG. 6.—Permeability Tests on Muscovite Mica and Clays.



| Curve | Investigator | Permeate | Permeant | $n_0$ | $D_E$ mm |
|---|---|---|---|---|---|
| 1 | Michaels and Lin | Kaolinite | Methanol | 0.483 | $9.52 \times 10^{-4}$ |
| 2 | " | " | Water | 0.544 | 10.33 " |
| $3_A$ | " | " | Nitrogen | 0.547 | 8.73 " |
| $3_B$ | " | " | Nitrogen | 0.568 | 8.00 " |
| 4 | " | " | Cyclohexane | 0.568 | 6.67 " |
| 5 | Howe and Hudson | Quartz Sand and Clayslip | Air | 0.173 | $3.64 \times 10^{-1}$ |

FIG. 7.—Permeability *versus* Porosity.



| Curve | Investigator | Permeate | Permeant | $n_0$ | $D_E$ mm |
|---|---|---|---|---|---|
| 1 | Lamb | Sand | Water | 0.273 | $1.60 \times 10^{-1}$ |
| 2 | Franzini | Sand | Water | 0.312 | " " |
| 3 | Taylor (After Gilboy) | Clay | Water | 0.425 | $4.57 \times 10^{-3}$ |
| 4 | " | Clay | Water | 0.453 | 3.62 " |

FIG. 8.—Permeability *versus* Porosity.

expresses the interaction at the fluid-solid interface.

If these assumptions are correct and we plot the permeability factor $k$ as a function of $n$ according to Eq 8, we will get a straight line intersecting the abscissa at $n = n_0$ and having a slope of $\tan A = \frac{\gamma}{32\,\mu} D_E^2$ (Fig. 5). Experimental results agree remarkably well with our theoretical Eq 8 and the above assumptions. Figure 6 shows results from tests run in the Soil Mechanics Laboratory of Princeton University.

During our investigation, an extensive literature search was conducted in order to find existing data which might be evaluated in the light of our Eq 8. The vast majority of these papers could not be used because they covered only a very narrow range of porosities or were lacking in essential test data or because their results were presented in a form too tedious to evaluate (Blake's method of plotting dimensionless groups). Also, some reported results just did not make sense and thus indicated that some controlling factors or conditions of the test had probably been overlooked.

A few of the papers were very valuable. Figure 7, for example, shows the results of Michaels and Lin (**24**) as well as data from an experiment by Howe and Hudson (**17**). In Fig. 8, data reported by Lamb (**20, 21**) and Taylor (**29**), as well as some results obtained by Franzini (**12**), are used.

It will be noted that all results deviate from the straight line in the range $n \approx n_0$, that is, where the total porosity approaches the ineffective porosity. This was expected, since here our assumptions that $D_E$ and $d_s$ are constants may be gross approximations only. Also, the type of flow probably changes gradually at these low porosities such that Darcy's law breaks down.

## THE DIAGRAM ln *k* *versus* VOID RATIO *e*

One may ask how Eq 8 can be reconciled with an observation made by several investigators (**20, 29**), namely, that, for a reason hitherto unexplained, the plot of the natural logarithm of $k$ *versus* $e$ ($e$ = void ratio) usually gives a straight line. This fact is not disputed and is exactly in line with our results.

TABLE I.—ERROR MADE BY NEGLECTING HIGHER ORDER TERMS.

| $n$ | $e$ | $e + \frac{1}{2}$ | $\ln n$ | $\frac{1}{\ln n}$ | Difference | Per Cent Error |
|---|---|---|---|---|---|---|
| 0.3..... | 0.43 | 0.93 | 1.2 | 0.83 | +0.10 | +10.7 |
| 0.4..... | 0.66 | 1.16 | 0.91 | 1.10 | +0.06 | +5.2 |
| 0.5..... | 1.0 | 1.50 | 0.69 | 1.45 | +0.05 | +3.3 |
| 0.6..... | 1.5 | 2.00 | 0.57 | 1.96 | +0.04 | +2.0 |
| 0.7..... | 2.33 | 2.83 | 0.36 | 2.81 | +0.02 | +0.7 |
| 0.8..... | 4.0 | 4.50 | 0.222 | 4.51 | −0.01 | −0.25 |
| 0.9..... | 9.0 | 9.50 | 0.105 | 9.52 | −0.02 | −0.2 |

*Relationship Between Void Ratio e and Porosity n:*

The logarithmic function $\ln\left(\frac{x+1}{x}\right)$ can be expanded for $x > 1$ into the series:

$$\ln\left(\frac{x+1}{x}\right) = 2\left[\frac{1}{2x+1} + \frac{1}{3(2x+1)^3} + \frac{1}{5(2x+1)^5} + \cdots\right]$$

Neglecting the higher order terms, we may write:

$$\ln\left(\frac{x+1}{x}\right) \approx \frac{2}{2x+1}$$

and set the void ratio $e = x$. Since $n = \frac{e}{1+e}$, then

$$\ln\left(\frac{e+1}{e}\right) = \ln\frac{1}{n} = -\ln n \approx \frac{2}{2e+1}$$

or:

$$e + \tfrac{1}{2} \approx -\frac{1}{\ln n}$$

which means that the relationship between $e$ and $n$ is logarithmic and hence a plot ln $k$ *versus* $e$ is actually a plot of ln $k$ *versus* ln $n$ and hence should be a straight line. Table I shows the error made by neglecting the higher order terms in the series for values within the practical range for $n$. As we can see from the last column of Table I, it is rather small.

## REVIEW OF PERTINENT LITERATURE

The idea that only part of the void space in a granular medium contributes to the flow during permeation is by no means new. Slichter (**28**) emphasized the importance of the full porosity when he derived his expression for the equivalent channels in a permeate of equally sized spheres. King (**18**) mentions the term "effective porosity" and postulated that increase in rate of flow increases the effective porosity $n_E$ which is less than the true porosity. Sven Erikson (**9**) reports an experiment where he injected a salt solution into water flowing through a porous bed and measured the time for the salt to appear at the outlet. Assuming that this time would give him the actual interstitial velocity $U_i$ and that the effective porosity $n_E = \frac{U}{U_i}$ (where $U$ is the filter velocity), he found that, on the basis of his assumptions, the effective porosity increased steadily from 0.14 to 0.47, where it remained constant. He then advanced the hypothesis that a stationary film of fluid is retained on the surface and decreases as the rate of flow rises. A similar suggestion was offered by Darapsky (**6**), who surmised that a stationary liquid ring is retained at the point of each particle-contact and that its size is controlled by the velocity of flow. Fair and Hatch (**11**), who made an extensive investigation of the permeabilities of uniform filter sands, state in their paper: "... actually all the open area does not contribute to flow. There are stagnant spaces within channels of flow, side pockets and dead ends ...," but then proceed to induct an equation using the total porosity.

Baver (**1, 2**) also realizes that the porosity is only partially effective in transmitting flow and calls this part the noncapillary porosity. He and Winterkorn (**31, 32**) are the only investigators who tried to take into account merely part of the porosity for the determination of $k$. To evaluate the effective part, Bauer introduces a "porosity factor" as a function of the drained pore space under varying amounts of capillary tension and relates it to a "flex-point." His theory requires rather cumbersome experimentation and evaluation, and the physical significance and relation of this porosity factor cannot be readily understood.

TABLE II.—INTENSITY OF WATER FIXATION FORCES, KG PER SQ CM (AFTER WINTERKORN).

| | |
|---|---|
| First molecular layer | 25 000 |
| Hygroscopic water | 50 |
| Permanent wilting point | 12.5 |
| Wilting point (dead water) | 6.25 |
| Vacuum moisture equivalent | 0.55 |

One of the more lucid treatments of the permeation problem was presented by Winterkorn (**31**). He clearly realized that any concept concerning the permeability of soils must be reconcilable with the Hagen-Poiseulle equation as well as with experimental results. Winterkorn also recognizes the importance of the immovable "water" fixed at the liquid solid interface and gives the intensity of the water fixation forces (Table II). We think it necessary, however, to elaborate here that the ineffective porosity $n_0$ is not just the liquid volume fixed by the surface forces. In fact the larger portion of the ineffective porosity will be due to the liquid locked up in the channels with a "closed valve", the liquid locked up in the cross-capillaries and in the boundary layer and only a minor part will be contributed by the fixed layer itself.

Winterkorn's derivation and investigations were concerned with the permeability relationships during volume changes (swelling, slaking and consolidation of clays) that is according to our definitions with unstable permeation systems. Therefore our results are actually not comparable. However, it is interesting to note that he also found a "limiting or specific porosity" (**32**) in analogy to our $n_0$ of Eq 8 below which Darcy's law breaks down.

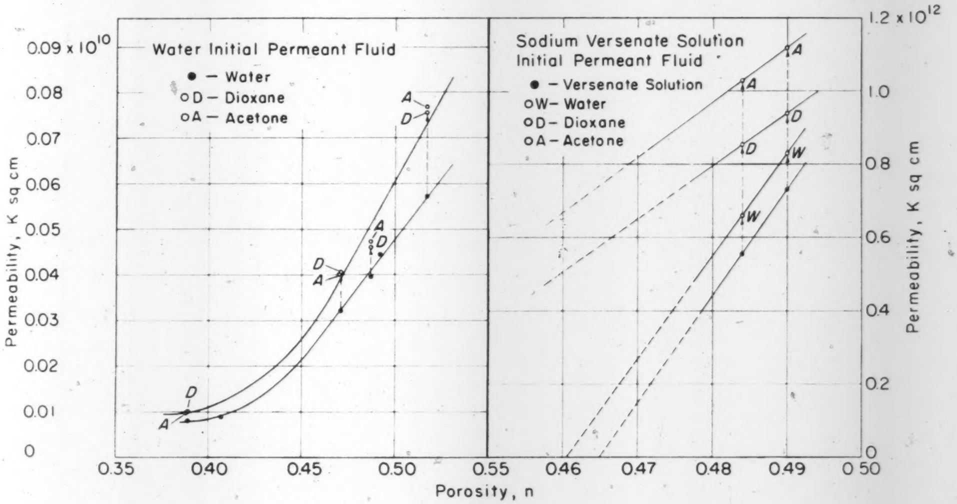


FIG. 9.—Effect of Desolvation on Kaolinite Permeability.

Winterkorn also states that, since the degree of fixation is an inverse power function of the distance from the wall, the volume of immovable "water" decreases with increasing hydrostatic pressure. This is important and partially explains the flare out of the velocity profile near the "surface" of the stationary layer (curve $C$, Fig. 1($b$)). Our concept of the stationary boundary-layer shares one difficulty with the classical boundary-layer concept, namely, that of defining its depth $d_s$, which always will be more or less arbitrary. Nature makes no twists as assumed at point $a$ in curve $B$, Fig. 1($b$). The true velocity profile will approach zero asymptotically. We propose to define the point $a$ as the point of maximum concave curvature of the velocity profile. This point then determines the thickness $d_s$ of the stationary boundary-layer.

Tests by Bozza and Secchi (**3**) and Michaels and Lin (**24**) (Figs. 7, 8, 9) clearly demonstrate that there is interaction between the liquid and solid surface exemplified by the different values $n_0$ for the same permeate (kaolinite) and different permeants. The reasoning is as follows: The degree of interaction (fixation) between permeate and permeant will determine the thickness of the stationary boundary-layer $d_s$. Again using the analogy of the pipe with a closed valve, a decrease in $d_s$ will open up some of the "valves" and hence diminish the ineffective porosity $n_0$ and thus increase the permeability. An increase in $d_s$ must have the reverse effect.

An analogous consideration will explain the rapid decrease in permeability when gaseous matter leaves the permeant (unstable permeation system). Here another interaction surface (liquid-vapor) is created. Once a gas bubble has formed it will be rather stable, first, because of the surface tension of the liquid and, second, because of the adsorbed molecular ionic layer of the vapor molecules at the liquid-gas interface. If such a bubble is swept against an already partially closed "valve" it will seal off the passage completely, thus rapidly decreasing the effective porosity.

Michaels and Lin (24) conducted very careful and interesting permeability experiments over a wide range of porosities, and their report deserves careful attention here. They measured the permeability of kaolinite to various permeants where the initial permeant was also the molding liquid. Their results were presented in plots of $k'$ *versus* $\frac{e^3}{1+e}$ where $k'$ corresponds to our permeability factor $k\frac{\mu}{\gamma}$ and hence has the dimension sq cm. The term $\frac{e^3}{1+e}$ is the porosity function of the Kozeny-Carman formula. According to this formula the diagram should be a straight line. The results, however, show clearly the inadequacy of this concept. Michaels and Lin's data were used to plot $n$ *versus* $k'$ in Fig. 7, giving excellent agreement with Eq 8. Another point which is brought out in their experiment is the influence of the dispersion of the permeate on the permeability. Michaels and Lin displaced the initial molding liquid and permeant by successive different permeants (Fig. 9). These results clearly show the change in the thickness $d_s$ of the adsorbed, stationary layer, which is indicated by the change in the ineffective porosity $n_0$, when the molding liquid was an aqueous, highly polar, dispersing liquid. They established quite definitely that these "secondary" permeabilities never approached the primary ones where the permeant was also the molding liquid.

In conclusion we would like to comment briefly on two points. The first one concerns the usage of terminology.

As we have shown there is nothing constant in the permeation problem except the ineffective porosity $n_0$ and possibly the effective diameter $D_E$ at porosities not in the neighborhood and below $n_0$. It therefore is not logical to use the term constant. The same objection must be raised against "coefficient of permeability." The permeability factor, as we propose it should be called, is a property of a permeate-permeant system and is neither a constant nor a dimensionless coefficient, but rather it is determined by the unit weight and viscosity of the liquid, the effective diameter $D_E^2$ of the permeate, and the effective porosity $n - n_0$ where $n_0$ describes the interaction between permeate and permeant (Eq 8). It will have the dimension cm per sec.

The second point concerns the use of consolidation data for the investigation of permeability relationships.

Here a word of caution is in order. Terzaghi's theory of consolidation was derived for the purpose of transposing experimental data from laboratory consolidation tests into settlement forecasts. In his derivation, Terzaghi assumed (*a*) that the permeability of the soil remains constant and (*b*) that the amount of expelled pore water remains constant for equal time intervals throughout the consolidation process. The general validity of his theory in transposing laboratory data into settlement forecasts is undisputed and has been proved in over 30 years of successful application. Observing Figs. 5, 6, 7 and 8, however, we note that the permeability varies rapidly with only slight changes in porosity. Also, while the theory is extremely successful in determining the magnitude of settlements, predicted and observed settlement rates —where the permeability comes into play—often differ considerably. This is then always explained by the anisotropy of the clay layers (different permeabilities in horizontal and vertical direction). Whatever the cause may be, it appears to us rather hasty to accept hypothetical assumptions which we know can be only approximately true and about whose degree of approximation very little or, actually, absolutely nil is known. While we do not question the indirect determination of the permeability from consolidation test data as an acceptable method of finding the over-all properties of a soil deposit on a semi-quantitative basis, we must challenge the use of this method in conducting research on the factors which influence the permeability of stable systems. We have to make enough assumptions as it is. Any additional ones will only magnify the errors of approximation in unknown proportions. Hence, in research on permeability, only methods which measure the permeability factor directly should be employed.

## SUMMARY

We hope to have shown that experimental results on the permeability of granular media can be reconciled with the theory of viscous laminar flow. Like any new concept, our concept of considering the adsorbed film as a stationary boundary-layer—while answering a number of previous open questions—immediately poses new ones. Some time-honored concepts and formulae may require reappraisal and modification, among others for example, the Kozeny-Carman formula and Stokes' law. The adequacy of the former must be drawn into serious doubt.

Stokes' law, expressing the velocity of a sphere settling in a liquid of infinite extent, has never given results agreeing with experiment whenever the diameter of such "spheres" was microscopically small and the liquid highly polar. This has always been attributed to the shape-factor, the mutual interference of adjacent particles in the hydrometer analysis and to different specific gravities of individual particles. While all these factors certainly do come into play, we believe that the adsorbed stationary boundary-layer plays a major role here also. An attempt to consider the influence of adsorbed layers for a modification of Stokes' law was made by Winterkorn and Moorman (**35**).

The adsorbed stationary boundary-layer is a solid-liquid interface phenomenon. The science of surface phenomena is still in its infancy and little is known about the origin, nature, and extent of surface forces. It would lead us too far astray to discuss the application of the stationary boundary-layer concept with all its ramifications and the reader is referred to the pertinent literature on surface properties (**14,23,36**). But we believe that the greatest value of the stationary boundary-layer principle will probably be realized in the investigation of surface phenomena as it will allow the experimental determination of the thickness of adsorbed layers.

## CONCLUSIONS

1. The fundamental concept of the stationary boundary-layer brings about excellent agreement between the theory of viscous flow and experimental results on the permeability of granular media.

2. The principle of a stationary boundary-layer is universally applicable wherever viscous laminar flow has to be investigated on a microscopic scale. It should be particularly valuable in the investigation of surface phenomena.

3. The permeability factor for porous media in steady and stable permeation is

given by Eq 8, where the ineffective porosity $n_0$ is constant and the effective diameter $D_E$ is constant for $n > n_0$.

4. The ineffective porosity $n_0$ can be clearly defined as the intercept on the abscissa of the extended straight line in the *n-k* diagram. It expresses the interaction between permeate and permeant.

5. The ineffective porosity $n_0$ is a function of the surface energy of the solids, the topography of the surfaces, the granulometry of the permeate, the particle orientation and dispersion of the permeate, and the polarities of permeant and molding liquid.

6. In the range $n \approx n_0$ and below, additional phenomena occur which need further clarification.

*Acknowledgments:*

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## REFERENCES

(1) L. D. Baver, "Soil Porosity in Relation to Gaseous and Water Movement," *Transactions*, Am. Geophysical Union, Part 2, pp. 414–419 (1940).

(2) L. D. Baver, "Soil Physics," John Wiley & Sons, New York, N. Y. (1940).

(3) J. Bozza and B. Secchi, *Gazetta Chimica Industria*, Vol. 11, pp. 443–487 (1929).

(4) P. C. Carman, "Fluid Flow Through Granular Beds," *Transactions*, Inst. Chemical Engrs. (London), Vol. 15, pp. 150–166 (1937).

(5) T. H. Chilton and A. P. Colburn, "Pressure Drop in Packed Tubes," *Journal Industrial and Engineering Chemistry*, Vol. 23, pp. 913–918 (1931).

(6) J. Darapsky, *Zeitschrift für mathematische Physik*, Vol. 60, p. 170 (1912).

(7) H. Darcy, "Les Fontaines Publiques de la Ville de Dijon," Paris (1856).

(8) A. J. E. J. Dupuit, "Etudes Theoriques et Pratiques sur le Mouvement des Eaux" (1863).

(9) Sven Erikson, *Journal für Gasbeleuchtung* Vol. 63, p. 615 (1920).

(10) Estermann and O. Stern, *Zeitschrift für Physik*, Vol. 61, p. 95 (1929).

(11) G. M. Fair and L. P. Hatch, "Fundamental Factors Governing Streamline Flow of Water Through Sand," *Journal*, Am. Water Works Assn., Vol. 25, No. 11, pp. 1551-1565 (1933).

(12) J. B. Franzini, "The Effect of Porosity on Permeability in the Case of Laminar Flow Through Granular Media," Ph.D. Dissertation, Stanford University (1949).

(13) H. J. Fraser, "Experimental Study of Porosity and Permeability of Clastic Sediments," *Journal of Geology*, Vol. 43, Dec., 1935, pp. 910–1010.

(14) R. Gomer and C. S. Smith, "Structure and Properties of Solid Surfaces," University of Chicago Press (1953).

(15) L. C. Graton and H. J. Fraser, "Systematic Packing of Spheres with Particular Relation to Porosity and Permeability," *Journal of Geology*, Nov.–Dec., 1935, p. 785.

(16) G. Hagen, "Ueber die Bewegung des Wassers in engen zylindrischen Röhren," *Pogg. Annalen*, Vol. 46, p. 423 (1839).

(17) W. L. Howe and C. J. Hudson, "Studies in Porosity and Permeability Characteristics of Porous Bodies," *Journal Am. Ceramic Soc.*, Vol. 10, p. 443 (1927).

(18) F. H. King, "Principles and Conditions of the Movements of Ground Water," *19th Annual Report*, U. S. Geological Soc. (1899).

(19) Josef Kozeny, *Sitzungsberichte der Wiener Akademie der Wissenschaften*, Vol. 136, Part 2(*a*), p. 271 (1927).

(20) T. W. Lamb, "Soil Testing for Engineers," John Wiley & Sons, Inc., New York, N. Y. (1951).

(21) T. W. Lamb, "The Permeability of Fine Grained Soils," Symposium on Permeability of Soils, p. 56, Am. Soc. Testing Mats. (1954). (Issued as separate publication *ASTM STP No. 163.*)

(22) T. W. Lamb, "The Storage of Oil in an Earth Reservoir," *Journal Boston Soc. Civil Engrs.*, Vol. 43, No. 3, July, 1956, p. 179.

(23) H. Mark and E. J. W. Verwey, "Advances in Colloid Science," Vol. III, Interscience Publishing, Inc., New York, N. Y. (1950).

(24) A. S. Michaels and C. S. Lin, "The Permeability of Kaolinite," *Industrial and Engineering Chemistry*, Vol. 46, June 1954, pp. 1239–1246.

(25) J. Poiseulle, "Recherches Experimentales sur le Mouvement des Liquides dans les Tubes de Très Petits Diamètres," *Comptes Rendus*, Vol. II, pp. 961, 1041 (1841).

(26) L. Prandtl, "Ueber die Flussigkeitsbewegung bei sehr kleiner Reibung," *Proceedings*, III, Internat. Mathematics Congress, Heidelberg (1904).

(27) H. Schlichting, "Boundary Layer Theory," McGraw-Hill Book Co., Inc., New York, N. Y. (1955).

(28) Charles S. Slichter, "Theoretical Investigation of the Motion of Ground Water," *19th Annual Report*, U. S. Geological Soc. (1899).

(29) T. W. Taylor, "Fundamentals of Soil Mechanics," John Wiley & Sons, Inc., New York, N. Y. (1948).

(30) Charles Terzaghi, "Determination of Permeability of Clay," *Engineering News-Record*, Vol. 95, pp. 832–836 (1925).

(31) H. F. Winterkorn, "Water Movement Through Porous Hydrophylic Systems under Capillary, Electrical and Thermal Potentials," Symposium on Permeability of Soils, p. 27, Am. Soc. Testing Mats. (1954). (Issued as separate publication *ASTM STP No. 163.*)

(32) H. F. Winterkorn, "Mechanism of Water Attack on Dry Cohesive Soil Systems," *Soil Science*, Vol. 54, No. 4, Oct., 1942.

(33) H. F. Winterkorn and L. D. Bauer, "Sorption of Liquids by Soil Colloids," *Soil Science*, Vol. 38, No. 4, Oct., 1934; Vol. 40, No. 5, Nov., 1935.

(34) H. F. Winterkorn, "Studies on the Surface Behavior of Bentonites and Clay," *Soil Science*, Vol. 41, No. 1, Jan., 1936.

(35) H. F. Winterkorn and R. B. Moorman, "A Study of Changes in Physical Properties of Putnam Soil Induced by Ionic Substitution," *Proceedings*, Highway Research Board, Vol. 21, pp. 415–434.

(36) Symposium on Exchange Phenomena in Soils, Am. Soc. Testing Mats. (1952). (Issued as separate *ASTM STP No. 142.*)

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## DISCUSSION

MR. W. G. HOLTZ.[1]—These comments were prepared by Mr. C. W. Jones of the Bureau of Reclamation.

The author has presented his subject in a clear and direct manner. The development of his theory is in line with the current trend of thought toward considering the active properties and influence of the smaller soil particles on the behavior of the soil mass.

We in the Bureau of Reclamation have noticed the difference between permeability theory and actual results. This has led us to sometimes adopt empirical factors to assist in correlations between the two. Perhaps analyses, such as those offered by the author, will aid us in an understanding of what actually causes the difference between theory and actuality on this subject.

The difficulty of applying theory such as the author presents to actual field permeability and seepage problems is that it is usually not possible to make assumptions of stable conditions such as he makes, at least in some of our work. For example, in the seepage from unlined or earth-lined canals, the temperature gradient is constantly changing, causing variation in fluid viscosity and deposition or absorption of air in the soil. Also, there is usually ionic exchange occurring and the degree of saturation of the soil is subject to change during the irrigation season. In most earth dams the reservoir level varies with time. More stable flow conditions undoubtedly do occur at depth in large hydraulic earth structures or in foundations.

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Although such variations as those mentioned above do occur, we should nevertheless not let them deter us from basic permeability studies such as the author has accomplished. Possibly, after stable flow has been more thoroughly investigated, the variations occurring in the field can be taken into account in at least an average or approximate manner.

MR. HAROLD W. OLSEN[2] (*by letter*).—The author has proposed a new permeability equation that is based on a model of viscous flow through capillary tubes and the concept of a fixed boundary layer. He has presented experimental data on mica, kaolinite, and sand which, he claims, support the permeability-porosity relationship that is predicted by his equation. On this basis, he concludes: "The fundamental concept of the stationary boundary layer at last brings about excellent agreement between the theory of viscous flow and experimental results on permeability of granular media."

It is questionable whether the data presented actually agree with the permeability-porosity relationship of the author's equation, and whether his equation is an improvement over the well-known Kozeny-Carman equation.

The author points out in his literature review that Michaels' and Lin's permeability data on kaolinite fail to plot linearly with the porosity function $n^3/(1 - n)^2$. Since the Kozeny-Carman equation predicts that such plots should be linear, the author concludes that this equation is inadequate. He continues then to say, "Michaels' and Lin's data were used to plot $n$ *versus* $k'$ in Fig. 7, giving excellent agreement with our Eq 8."

From Fig. 10 a direct comparison can be made between these equations concerning the degrees to which they agree with Michaels' and Lin's data on kaolinite permeated with water. The same data are plotted twice; permeability is plotted *versus* the porosity function $n^3/(1 - n)^2$ in the lower plot, and *versus* the porosity $n$ in the upper plot. The axes and the data in the upper plot are the same as the author's Fig. 7, curve *2*, except that in Fig. 10 the abscissa scale is expanded much more than that in the author's Fig. 7. This expansion provides a clearer picture of the experimental permeability-porosity relationship, and it is valid since adequate and sufficiently precise data exist to define the curve. Figure 10 shows that the data are not quite linear in the plot of $k'$ *versus* $n^3/(1 - n)^2$. However this degree of linearity is greater than that in the upper plot of $k'$ *versus* $n$. If, as the author states, the Kozeny-Carman equation is inadequate, it appears that his equation is even less adequate.

Figure 11, similarly, compares the degrees to which the equations agree with data on sand. The data for curve *1* in both plots are the same data as the author used in his curve *1* of Fig. 8. The data for curves *2* and *3* were obtained in the Massachusetts Institute of Technology's soil mechanics laboratory. The figure shows that the data are approximately linear for both porosity functions.

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[2] Graduate Student in Soil Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

Hence for these data on sands, both equations appear equally valid.

It is also questionable whether the theoretical basis of the author's equation is as valid as that of the Kozeny-Carman equation. Both equations are derived from Poiseuille's law for viscous flow through capillary tubes and from Darcy's law for flow through porous media. However, they differ in several respects. The author includes in his equation the boundary layer concept which is not in the Kozeny-Carman equation. In addition, the equation models differ in the way capillary tubes are used to represent the size, number, and length of pores in porous media. These latter differences are:

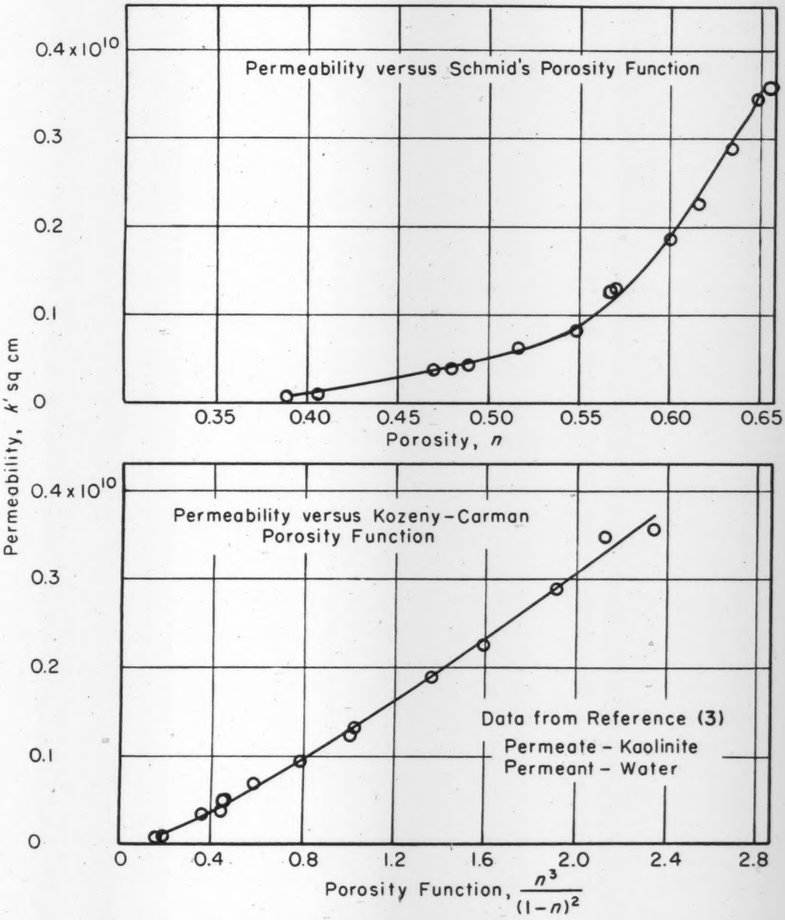

FIG. 10.—Permeability of Kaolinite *versus* Schmid's and Kozeny-Carman's Porosity Functions.

1. The length of capillary tubes in the author's model are set equal to the soil mass length; whereas in the Kozeny-Carman model, they are set equal to the length of tortuous flow paths through the soil.

2. Soil porosity changes are represented in the author's model by the

variation of the number of constant diameter capillary tubes per unit area; whereas in the Kozeny-Carman model, they are represented by the variation of the diameter and spacing of a constant number of capillary tubes.

The following discussion of the successes and shortcomings of the Kozeny-Carman equation will provide a basis for evaluating the validity of its model. The differing aspects of the author's model can then be examined to see if they are an improvement over those of Kozeny-Carman's model.

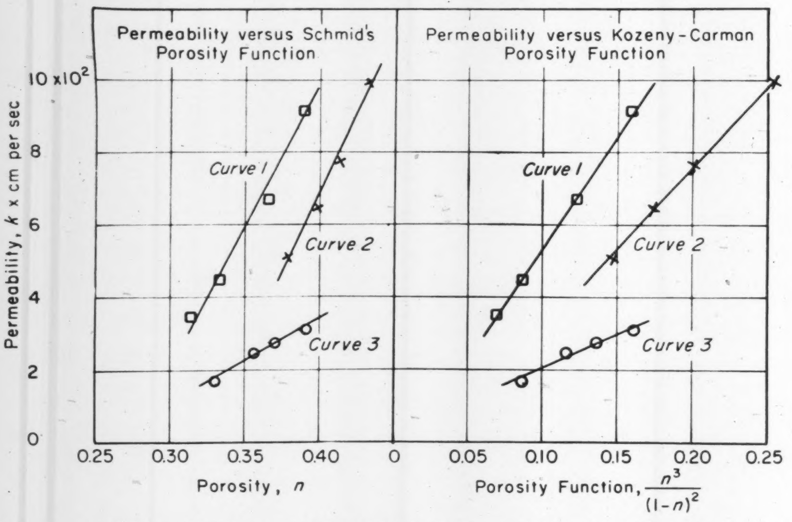

FIG. 11.—Permeability of Soils *versus* Schmid's and Kozeny-Carman's Porosity Functions.

Since the Kozeny-Carman equation appeared in 1937, extensive investigations have been conducted to ascertain the extent of its validity. Carman, in his recent book,[3] has analyzed much of the accumulated data, and presents evidence therein to show that measured permeabilities agree closely with permeabilities computed from the Kozeny-Carman equation for porous media which conform to the theoretical assumptions of the equation. These assumptions are, that all pore sizes are uniform and the particles are randomly packed. These conditions hold for uniform sands and silts, but they are violated for clays and micas in which the anisometric particles do not pack randomly, and well graded granular soils in which a range of pore sizes exist. Loudon's studies[4] contain further evidence that this equation is closely valid for clean sands. His measured permeabilities agreed within 20 per cent of those computed from the Kozeny-Carman equation.

Studies have been made to ascertain what factors cause discrepancies between measured and computed (from the Kozeny-Carman equation) permeabilities in clays.[5] The factors fall into two general categories: interfacial phenomena (the influence of electrical forces concentrated at the liquid-solid interfaces that act on the permeating fluid), and particle packing characteristics (the degree of particle dispersion and particle orientation). Michaels' and Lin's studies on kaolinite were directed towards determining the relative importance of the effects of these factors.[5] They concluded that particle packing characteristics were primarily responsible, and that the effects of interfacial phenomena were minor. They further concluded that the effects of interfacial phenomena could be attributed primarily to counter electroosmosis,[6] and consequently that the actual thicknesses of immobilized liquid films on the kaolinite particles must be extremely small, less than 4 per cent of the diameter of the pores. This conclusion, that immobilized liquid films actually are very small, has been further supported by recent evidence from studies on adsorption by Martin,[7] and from studies on heats of wetting by Rosenquist.[8]

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[3] P. C. Carman, "Flow of Gases Through Porous Media," Academic Press, Inc. (1956).

[4] A. G. Loudon, "The Computation of Permeability from Simple Soil Tests," *Geotechnique*, Vol. 3, p. 165–183 (1952).

[5] A. S. Michaels and C. S. Lin, "Permeability of Kaolinite," *Industrial and Engineering Chemistry*, Vol. 46, June 1954, pp. 1239–1246.

Since measured and computed permeabilities agree closely for porous media which conform to the assumptions of the Kozeny-Carman equation, the way in which the equation model represents their pore structure must be a close analogy. Consequently, it appears that the path a fluid particle follows through the soil is tortuous, and the diameter of pores vary with porosity approximately according to the relationship in the Kozeny-Carman equation. Since the author's model differs in these respects, it seems to be a less valid analogue of the pore structure.

The difference in the porosity functions of the equations comes about because in Kozeny-Carman's model pore diameters vary with porosity, whereas in the author's model they remain constant. Hence, the considerations in the previous paragraph suggest that Kozeny-Carman's porosity function has the more realistic theoretical basis for clean uniform sands and silts. For fine grained soils as well, these features of the Kozeny-Carman model (tortuous flow paths, and pore diameter varying with porosity) might represent the soil pores more accurately than do those of the author's model. This contention is supported by the accompanying Fig. 10, since for kaolinite, agreement with the Kozeny-Carman porosity function was closer than that with the author's porosity function.

The author's equation contains an ineffective porosity term that has as its theoretical basis the stationary boundary layer concept. The author concludes that this term "is a function of the surface energy of the solids, the topography of the surfaces, the granulometry of the permeate, the particle orientation and dispersion of the permeate, and the polarities of permeant and molding liquid." These factors are essentially the same as those proposed to explain the shortcomings of the Kozeny-Carman equation for clays, interfacial phenomena and particle packing characteristics.

Strictly speaking, the ineffective porosity in the author's model is the immobile fluid adjacent to the capillary tube walls, and it represents in soil the immobile fluid adjacent to particle surfaces. The boundary layer concept certainly seems to be a reasonable analogy for representing immobilized liquid films. Is the concept reasonable, however, for representing the effects of counter electro-osmosis and particle packing characteristics? The following considerations suggest that it is not.

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[6] A. S. Michaels and C. S. Lin, "Effects of Counter Electro-osmosis and Sodium Ion Exchange on the Permeability of Kaolinite," *Industrial and Engineering Chemistry*, June 1955, p.1249–1253.

[7] R. T. Martin, "Water Vapor Sorption on Kaolinite: Hysteresis," presented at the 6th Nat. Clay Conference, Berkeley, Calif., Aug., 1957.

[8] I. Th. Rosenquist, "Investigations in the Clay-Electrolyte-Water System," *Publication No. 9*, Norwegian Geotechnical Inst. (Oslo), p. 92–100 (1955).

The rate of osmotic flow in a capillary tube is proportional to the square of the tube radius; whereas the rate of hydraulic flow, is proportional to the fourth power of the radius. It follows, then, that counter electro-osmotic flow will not be represented correctly in a relationship based on Poiseuille's law, such as either the author's or the Kozeny-Carman equation.

The ways in which particle packing characteristics influence permeability have been envisioned by Michaels and Lin as follows:

1. *Degree of particle dispersion.*—Soils having a low degree of dispersion consist of aggregates of particles, each aggregate acting hydrodynamically as a single particle. Varying degrees of particle dispersion in a soil may be envisioned as varying effective particle sizes, and hence, varying pore sizes available for flow.

2. *Particle orientation.*—As the degree of preferred alignment of anisometric particles increases in a direction perpendicular to the direction of flow, the length of path followed by a fluid particle increases and thereby decreases permeability.

Reasonable analogue concepts for these factors might be, an effective pore diameter that relates to the degree of particle dispersion, and a varying tortuosity of flow path that relates to the degree of particle orientation. The concept of a stationary boundary layer seems to have little relationship to these mechanisms.

In summary, the author's permeability equation does not appear to be validated by his experimental data, and its theoretical basis appears to be less valid than that of the Kozeny-Carman equation. The concept of a stationary boundary layer may be a useful analogy for taking into account the effects of immobile liquid films on permeability, but it is not analogous to the effects of counter electro-osmosis and particle packing characteristics which, according to Michaels and Lin, are by far the most significant factors.

MR. WERNER E. SCHMID (*author's closure*).—The comments made by Mr. C. W. Jones are well taken, since they point out the limitations of the theory presented. All too often, solutions of a problem are applied in situations which have absolutely no resemblance with the conditions for which these solutions were derived. The author agrees with Mr. Jones that before we can ever hope to understand a problem with all its ramifications, we must first understand the most basic and simple relationships.

That this is not yet everywhere the case is revealed by the discussion of Mr. Olsen. Mr. Olsen defends the Kozeny equation with arguments which are either based on error, or based on unsubstantiated facts. Also, some of the arguments suggest a fundamental misunderstanding of the Kozeny equation and a lack of familiarity with recent developments. For example:

1. His upper diagram of Fig. 10, as well as our Figs. 6, 7, and 8, reveal for the lower porosities a break down of Darcy's law or more specifically a break down of the assumption $k$ being independent of the pressure gradiant. A functional relationship between $k$ and gradiant $p$, in particular the existence of an initial pressure gradient, has been proved experimentally by Derjaguin and Krylov.[9] Theta plotted against the Kozeny porosity function do not reveal this fact (see Fig. 10 lower diagram).

2. There is wide disagreement on the thickness of the immobilized films on the particle surfaces. Some indirect evidence (heat of wetting) is pointing toward a relatively thin rigid layer in the neighbor-

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[9] B. V. Derjaguin and N. A. Krylov, "Works of the Conference on Viscosity of Liquids," Vol. 2, Science Press, USSR (1944).

hood of 4 to 6 molecule diameters or 10 Å, whereas other data and reasoning support the view that the immobilized rigid layers may be as high as several hundred Å. Here it should be pointed out that from studies on adsorption and heat of wetting, we can at best hope to get some information on the thickness of rigid films under static conditions. The fixed boundary layer, however, is a dynamic phenomenon which depends among other things also on the surface topography, wake effect, increase in layer thickness in the downstream direction as we have pointed out in the paper. Most of these have no or little effect on the heat of wetting or on adsorption. Also, the permeant may be adsorbed by the surface due to osmotic effects as described by Proctor, Proctor and Wilson according to Winterkorn (**32, 33**) and Mattson.[10] This osmotic type of hydration does not take place with the liberation of measurable quantities of heat. Incidentally, nothing could be more falacious than to claim as Mr. Olsen does, that the thickness of the immobilized layer is not larger than 4 per cent of the pore diameter. This percentage will vary with the pore diameter and can be any where from practically zero to 100 per cent.

3. Mr. Olsen claims that the Kozeny model must be more realistic, since there the pore diameter varies with porosity, whereas in ours it does not. This is wrong. The relations between pore diameter and porosity are given by our Eq. 7. Here, Mr. Olsen seems to have confused pore diameter of the model and the effective diameter. Our assumption that the *effective* diameter remains constant, implies the logical conclusion that the change in porosity is primarily sustained by the largest diameter pores and that it has practically no influence on the considerably smaller effective diameter. This assumption is well supported by the experimental evidence. Mr. Olsen's contension that our permeability equation "does not appear to be validated" by our experimental data and that the "theoretical basis appears to be less valid than that of the Kozeny-Carman equation" really cannot be taken seriously in view of the points mentioned above and in view of Figs. 6, 7 and 8.

4. The case against the Kozeny equation is stated most comprehensively by Adrian E. Scheidegger in his excellent recent book.[11] Although the book was not yet available at the time of our investigation, nor was Scheidegger aware of our results, the conclusions are identical. We quote from page 105 of his book:

> "An actual substantiation of the Kozeny equation is almost impossible because it contains three parameters ($S$, $c$, $T$), all of which would have to be determined by independent means in order to obtain a valid check. This, however, introduces many errors, especially in view of the fact that $T$ is not even properly defined as it was introduced by the Kozeny equation itself.
>
> "Nevertheless, a number of experiments have been performed which claim to "substantiate" the Kozeny theory, usually simply by measuring some of the constants occurring therein and showing that the values obtained are not unreasonable. Such experiments have been reported by Bartell and Osterhof (1928), Donat (1929), Kozeny (1932), Zunker (1932), Carman (1939), Wiggin, Campbell and Maass (1939), Fowler and Hertel (1940), Rigden (1943), Adams, Johnson and Piret (1949), Baline and Valis (1949), Kwong *et al* (1949), Rose and Bruce (1949), Coulson (1949, 1950), and Hoffing and Lockart (1951).
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> "An improvement over the above "verifications" of the Kozeny equation has been attempted by Wyllie and co-workers (Wyllie, 1951; Wyllie and Spangler, 1952;

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[10] S. Mattson, "The Laws of Soil Colloidal Behavior," *Soil Science*, Vol. 33, pp. 301–322 (1932).

[11] Adrian E. Scheidegger, "The Physics of Flow Through Porous Media," The Macmillan Co., New York, N. Y. (1957). University of Toronto Press, Toronto, Canada (1957).

Wyllie and Gregory, 1955). Here an attempt was made to attach a more precise physical meaning to the "tortuosity." Wyllie postulated that the electrical and fluid flow paths should be identical if the electrical and pressure potential were analogous. Thus, the tortuosity could be measured by independent means. However, inspection of Wyllie's work shows that there is some uncertainty regarding the proper expression of tortuosity in terms of the electrical analogue, reflecting itself in the fact that the Kozeny constant is, in every case, adjusted *a posteriori*.

"The Kozeny theory, therefore, contains some vague factors and it is the opinion of the writer that more than a qualitative decription of the phenomena cannot be expected from it."

and again on page 107 Scheidegger writes:

"Many discrepancies with the Kozeny theory can be obtained from experiments, provided not too many undetermined "fudge" factors are introduced into it. Thus, Kozeny himself (1927(*b*)), in an attempt to substantiate his formula experimentally, found discrepancies between calculated and measured surface areas of −69 to +86 per cent. It is a matter of taste whether one wants to consider these as merely experimental errors. More recently, Macey (1940) found enormous changes in permeability with porosity which are not explainable by the Kozeny equation. Similarly, Sullivan and and Hertel (1940) and Sullivan (1941) showed that Kozeny's law breaks down, at least in the case of highly porous, fibrous media. Walas (1946) found that a modification in the Kozeny equation is necessary if it is to be applied to filtration, and Adamson (1950) observed that the Kozeny constant has to be adjusted *a posteriori* in every experiment, which is, of course, quite unsatisfactory. Wyllie and Rose (1950), in an attempt to justify the Kozeny equation, place much emphasis upon the "tortuosity," which, however, is a rather vague physical concept, and, if measured electrically, is in need of a proper justification for an application to fluid flow. Furthermore, Brooks and Purcell (1952), again claiming that their results substantiate the Kozeny equation, in fact find a large discrepancy when areas are measured by the gas-adsorption method and with the aid of the Kozeny equation. Finally, Kraus and co-workers (Kraus and Ross, 1953; Kraus, Ross and Girifalco, 1953) attempted to correlate pressure drops and flow rates through beds of many materials with gross particle surface, as determined from geometric considerations, and with microstructure surface from nitrogen adsorption measurements. They found that both surfaces contribute to pressure loss, but by *different* mechanisms. Childs and Collis-George (1950) summarize the consequences of these experiments appropriately by stating that although most authors claim agreement with observed values of permeability, the fact seems to be that it has been impossible to secure a sufficiently wide range of variation of porosity and pore-size distribution to test the formulas severely, and the tests which have been reported above indicate a wide range of error. That the method of determining the specific area of powders by measurement of permeability has achieved considerable popularity may reflect only the essential similarity of most industrial problems involved.

"It seems indicated, therefore, that investigations of theories of permeability must be based upon assumptions other than the hydraulic radius theory."

These statements need no further elaboration.

# DEVELOPMENT OF A NEW CHEMICAL GROUT*

By R. H. Karol[1]

In contrast with other engineering materials, the properties of soil cannot be specified by the design engineer. Foundation loads must be designed to suit the soil, and the soil properties, and attendant foundation and excavation problems, depend only on the selection of a site. As industry grows and construction proceeds, the better construction sites (from a soils viewpoint) have been taken, and new construction must proceed on sites that become progressively more troublesome. The need for methods to modify soil properties becomes more and more acute, and consequently more and more attention and interest is being focused on materials and techniques for modifying the properties of soils.

## METHODS OF MODIFYING SOIL PROPERTIES

Soils consist of solid inorganic and organic discrete particles in surface contact with each other. The voids between particles are filled with fluid, usually air or water. In general, all methods of soil treatment can be divided into two categories: (*a*) changing the chemical nature of the soil particles, and (*b*) changing the soil voids. Most methods of soil stabilization fall into the latter category. These include well-pointing, electro-osmosis and vibroflotation, which reduce the void volume, and grouting, which changes the characteristics of the void fluid.

## DESIRABLE PROPERTIES OF A CHEMICAL GROUT

The most common material for grouting is cement. Cement grouting is an old and well established method, and many engineers are familiar with its uses and limitations. Sodium silicate is another chemical grout which has been known and used for many years. Considering the properties of these and other grouts it is simple to list the desirable properties for an ideal grout.

1. The grout must be able to modify soil properties as desired. In most cases two changes are desirable, (*a*) strength increase, and (*b*) permeability decrease. Although there are many cases where changing the soil properties for a short period is adequate, it is still desirable for most applications to have the effects of grouting last indefinitely. Therefore, the grout should cause a permanent increase in soil strength or a permanent decrease in permeability.

2. The grout must be a material which can be readily placed in the soil in sufficient quantities to do the job. This means that the grout must be applied as a liquid, since it is impractical to place dry chemical to any depth in a soil mass. Transporting a solution is expensive, and it is therefore desirable for the grout to be dry material, reaily soluble in water.

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* Presented at the Sixtieth Annual Meeting of the Society June 16–21, 1957.

[1] Soils Engineer, American Cyanamid Co., Princeton, N. J.

3. There must be a means for controlling the reaction time. There is always a time lapse between the mixing of the grout and its final location throughout the voids of a soil mass. Since the size of the mass to be treated and its permeability vary from job to job, the time lapse is generally different for each job. Thus it is highly desirable to be able to adjust accurately the time at which the stabilizing reaction takes place.

4. It is most desirable for the stabilizer solution to have a very low viscosity to minimize pumping problems, and it is also desirable that the viscosity of the solution remain unchanged until the stabilizing reaction takes place. In other words, the reaction must be capable of being inhibited, but it should be very rapid when it finally does occur.

5. The reaction should be relatively insensitive to the common impurities in water and soils, so that good field control can be obtained for the reaction time.

6. The chemicals should be noncorrosive so that they can be handled with common field piping and pumps.

7. The chemicals should be nontoxic and nonexplosive, so that personnel can safely handle them.

8. The materials should be inexpensive enough so that their use affords economic advantage over other means of producing the same end results.

Summarizing, the ideal chemical grout would be a cheap, effective soil solidifier, consisting of a water-soluble product which readily dissolves to form a nontoxic, noncorrosive solution of very low viscosity, whose reaction time can be readily and accurately controlled.

As yet, no one has discovered a material having every one of these properties.

## AMERICAN CYANAMID AM-9 CHEMICAL GROUT

Although no one has yet found the ideal grout, a number of chemicals have shown sufficient promise to warrant use in the field. One of the more recent products is known as AM-9 Chemical Grout. This chemical was first developed in 1951, with some of the original work being done at the Soil Stabilization Laboratories of MIT. Early field tests were conducted by Soil Testing Services Co. in Chicago.

AM-9 is a mixture of two organic chemicals, acrylamide and N,N′-methylenebisacrylamide, in proportions that produce very stiff gels from dilute aqueous solutions.

### *General Description:*

AM-9 is an extremely water soluble powder. Up to 50 per cent by weight can be dissolved in water fairly rapidly. Solutions of up to 30 per cent concentration are very stable and may be stored at room temperature for long periods of time. Such solutions have a very low viscosity, approaching that of water. Gels will form in concentrations as low as 2 or 3 per cent, although the properties of such gels will be erratic. Above 4 per cent concentration, gels with consistent reproducible properties will always form.

Formation of the gel is caused by the addition of small quantities of other chemicals, which catalyze the AM-9 solution. Although there are many catalyst systems which will cause gelation, ammonium persulfate (AP) and nitrilotrispropionamide (NTP) appear at this time to offer the most advantages.[2]

AM-9 has some toxic properties. Repeated contact with either the powder or a water solution by any route of exposure may affect the central nervous system. Such effects, however, gradually disappear if exposure is stopped. The use of a mask when handling the dry powder and the use of rubber gloves and coveralls when working with the solution have been found effective in elimination of the health hazard. To the best of present knowledge the final gel is not toxic except that it may contain small amounts of unreacted AM-9.

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[2] Recent work with $\beta$-dimethylaminopropionitrile (DAP) indicates that this chemical may be more effective and cheaper than NTP.

AP is, of course, an oxidizing agent, and care must be observed in handling it. The solution will discolor and possibly ruin clothing and may cause skin irritation. In addition, it will cause corrosion of iron and steel, so that it has been found desirable to use aluminum or stainless steel piping and vessels for field work. NTP offers no handling problems.

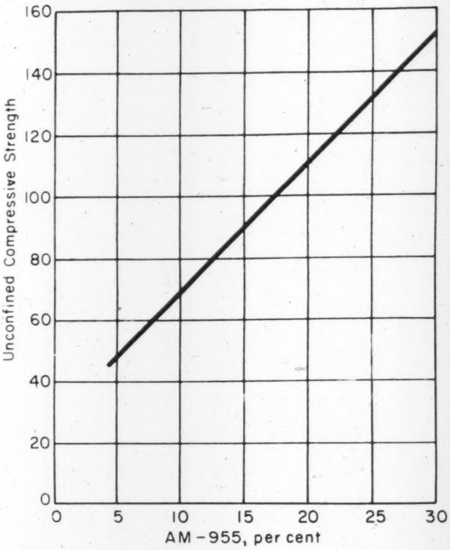

FIG. 1.—Unconfined Compression Tests on a Stabilized Fine Sand, Compacted to a Dense State.

### CHEMICAL PROPERTIES

Much of the initial chemical research carried out in the Stamford Laboratories of the American Cyanimid Co. was centered around finding catalyst systems that would permit adequate control of the induction period. In general, with a dual catalyst system, the two chemicals added combine to form a reaction product, R, and the formation of a gel is retarded until a sufficient concentration of R is present to cause catalysis.

The viscosity of the catalyzed solution, initially very near that of plain water, remains virtually unchanged during the induction period. When the concentration of R reaches a critical value, polymerization or gelation occurs very rapidly, and in a matter of a few minutes there is a change from a watery fluid to a rigid gel.

It has been found that within limits the induction time, that is, the time lapse between the addition of catalysts and the formation of gel, is independent of the concentration of AM-9.

Also within limits, it has been found that the gel strength is independent of the catalyst concentration and the resulting gel time, but varies with the concentration of AM-9. The gel is too plastic to permit tension or compression tests, but ball penetration tests have indicated that the strength *versus* AM-9 concentration relationship is linear.

### FIELD RESEARCH STUDIES

*Laboratory Work:*

One of the first discoveries to be made by the field research group was that under ordinary concentrations of AM-9 and catalysts, gels will not form in dry soils[3]. Apparently, in fine grained soils, capillary dispersion prevents the gel phase from being continuous, and as capillary effects decrease, gravitational effects increase, causing the injected solution to disperse by gravity flow. This meant that field work must be performed in soils that are saturated, or nearly so. Since many foundation problems are due to the uncovering of saturated sands and silts, this is by no means a serious drawback.

One of the first problems to be faced, and the question most often asked, is

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[3] Recent work with high catalyst concentrations has shown that gels can be made to form even in completely dry soils.

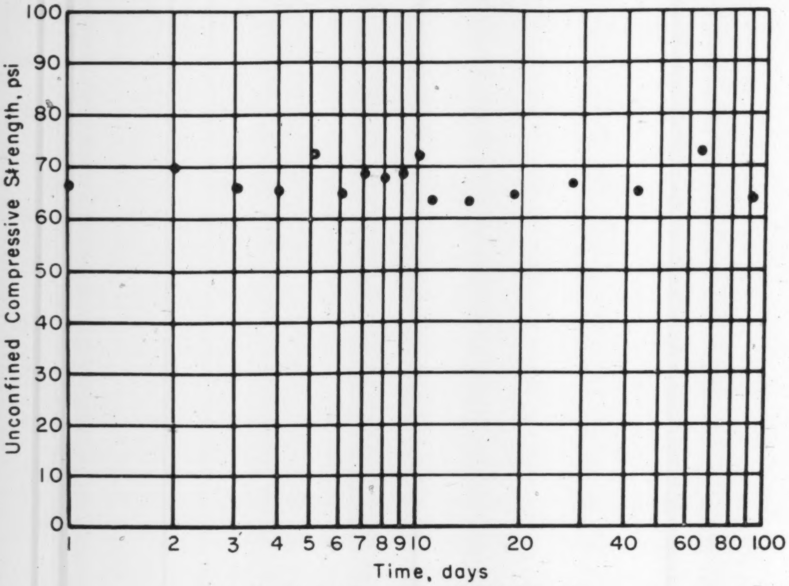

FIG. 2.—Aging Tests of Stabilized Sand.
Fine washed silica sand 10 per cent AM-955 solution stored in wet sand.

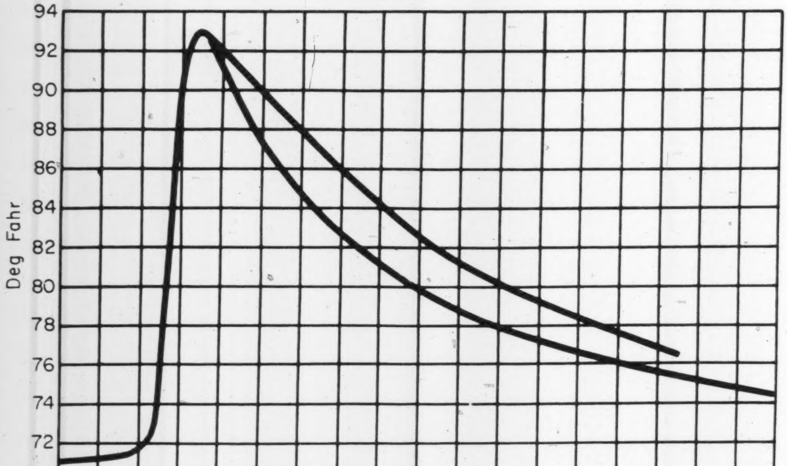

FIG. 3.—Chemical Activity Prior to and During the Gelation Reaction.
10 per cent AM-955, 0.5 per cent AP, and 0.25 per cent NTP.

how grain size affects the field injection work. The results of laboratory and field work have shown that grain size is not the only factor affecting pumping rates, and at the present time the best answer to this question is the following generalization:

Whenever the natural soil is such that sufficient water will flow through it to cause construction problems, it is usually possible to pump in enough chemicals in a reasonably short time to effect adequate stabilization. Effective stabilization jobs have been done in coarse and fine silts, and even in silts with some clay soil in them.

Another question often asked concerns the permeability of stabilized soil. Experience and experiments have shown that for all practical purposes a mass of stabilized soil is impermeable. However one additional factor must be considered. Under normal concentrations, the gel formed is 80 to 90 per cent water, contained within the gel matrix. When exposed to air or to excessive heat, this water will evaporate, creating voids in the stabilized soil mass. Dried out masses will no longer be impermeable. When submersed in water, a dried gel will reabsorb moisture and swell to about its original volume. However, the process of drying and wetting will cause shrinkage cracks in loose soil masses, and these will probably not seal with reabsorption of water. Thus, it is more nearly correct to say that a stabilized soil mass which is not permitted to dry out will remain impermeable.

In many construction problems it is desirable to increase the effective strength of the soil. A large number of unconfined compression tests have been performed, in the Princeton Laboratories and at Lehigh University, to determine the strength of stabilized soils. It has been found that the unconfined compressive strength varies directly with the amount of AM-9 in solution, as shown in Fig. 1.

This particular set of tests was performed on a narrowly graded fine sand, compacted to a relative density approaching 100 per cent.

It has also been found that for a given concentration of AM-9, strength increases with decreasing grain size and increases with increasing spread of grain size. Most of the tests performed including those shown in Fig. 1 were with freshly stabilized soils, that is, no drying has been permitted. As the soil dries, the strength increases, and a completely dried soil will show strengths approaching that of cement mortar.

The question of permanence is also of vital interest. Laboratory work has shown that once the gel is formed, it is pretty much there to stay. No solvents have been found that will destroy the gel and at ordinary temperatures it is extremely difficult to break down, even with strong acids and bases. To verify this, a series of tests was begun on a large number of stabilized soil cylinders. These were all fabricated to within 1 per cent of the same density, then stored under saturated sand to prevent drying. Periodically, several samples are tested in unconfined compression to determine if there is any change in strength. The results to date (Fig. 2) show:

No significant change in strength with aging. The scatter of test points are within the limits of reproducibility obtained on a large number of tests performed immediately after fabrication.

Although it was known that actual gelation in a solution of AM-9 occurs very rapidly, there was some question as to whether this would hold true for a solution spread through the voids in a soil. The process of gelation gives off heat, and the change in temperature is a good indication of chemical activity. This phenomenon was used to investigate the reaction in a soil mass. Figure 3 shows the temperature *versus* time relationship for a soil sample saturated with catalyzed AM-9 solution.

The very abrupt temperature rise indicates gelation, and the rapid falling off of temperature indicates that virtually the entire gelation reaction occurs within a very short period. This is verified by the shorter curve which represents the cooling rate of an uncatalyzed solution raised to the same maximum temperature as the gelled soil and air cooled under identical conditions.

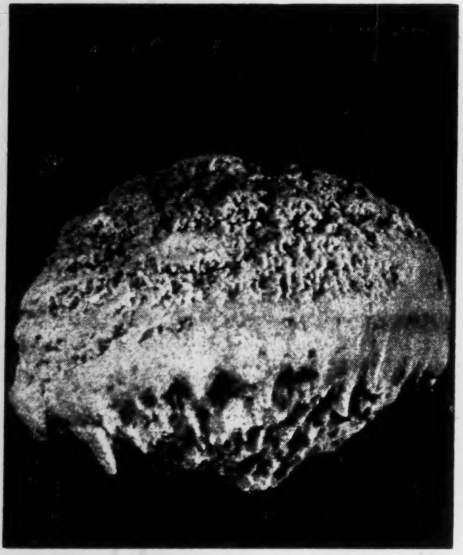

FIG. 4.—Typical Shape of Stabilized Soil Mass Obtained by a Single Injection into a Uniform Sand.

The curve is also a good representation of time-viscosity relationships. The first portion of the curve represents the initial viscosity of the solution, approximately equal to that of water. The peak of the curve represents the very high viscosity of the solid gel. As shown, the viscosity change from liquid to solid is almost instantaneous.

The size and shape of a soil mass stabilized by injection is of considerable interest. At first it was thought that the shape could be influenced by the location and spacing of the openings in the injection pipe. However, it has been found that, in a uniform soil, the shape of the stabilized mass is roughly spherical regardless of the size, shape, or spacing of the holes in the injection pipe. A typical ball of stabilized soil is shown in Fig. 4.

Considerable effort was spent in trying to establish ratios of stabilized volumes to injected volumes, with the final conclusion that laboratory results could not be extrapolated to field scale. This type of information is now being gathered from field experience, and will be discussed later.

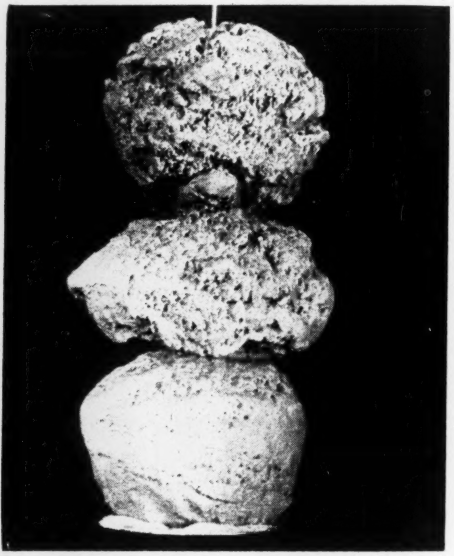

FIG. 5.—Interlocking Balls of Stabilized Soil Formed by Successive Vertical Injections.

Injection pipe was driven down a distance equal to the ball diameter between shots.

For field work, an obvious procedure is to drive the injection pipe further into the soil for subsequent injections of AM-9. The results of such a procedure were investigated in the laboratory. Some of this work is illustrated in Figs. 5 and 6.

In Fig. 5, 1000 ml of solution was injected three times, with the pipe driven 6 in. between shots. (The 6-in. driving distance was chosen since it was known that 1000 ml injection stabilizes roughly a 6-in. diameter sphere.) As can be seen, the separate injections are clearly outlined. The plane joining the balls is a plane of weakness.

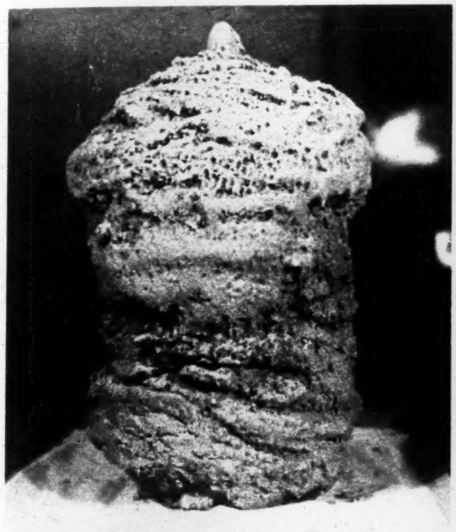

FIG. 6.—Stabilization of a Column of Soil by Successive Injections Spaced Vertically Apart a Distance Equal to the Distance of Radial Flow.

Figure 6 shows the result of the same experiment with the pipe driven only 3 in. between injections. Here a fairly uniform column of soil was stabilized, which was the desired result. This type of injection is applicable to caisson work and to well drilling through sands and silts.

From laboratory work with small scale injections a number of facts for field use were discovered. The most important of these are:

1. In uniform soils, the injected solution spreads radially from the point of injection.
2. For large non-uniform soil masses, new injections should not be made until previous injections have gelled.
3. For best continuity between adjacent injections, pipes should be spaced at about the distance of radial flow.

Manifolding of injection pipes appeared to be an obvious field procedure for reducing the time required for actual jobs. This procedure was investigated by laboratory work to determine its limitations.

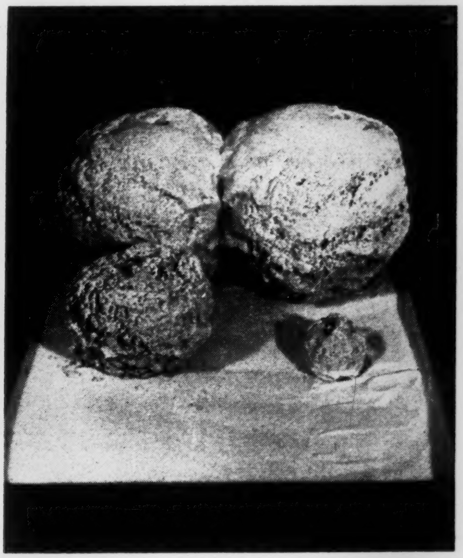

FIG. 7.—Stabilized Soil Masses Indicating Flow Through Uncontrolled Manifolded Injection Pipes.

Figure 7 shows the results of one such test. Here, four pipes were used, placed at the corners of a 6-in. square; 2000 ml of solution were injected through one hose, which was manifolded to the four pipes. This figure illustrates very clearly the major deduction from manifolded work. Although the soil was very uniform and all other conditions were maintained as uniform as possible, flow was still highly selective as indicated by the size differences between the stabilized masses. This has been verified many times by other experiments and leads to the conclusion that manifolding of pipes for field work should not be done without some means of controlling or metering the flow to each injection pipe.

Construction problems due to quick conditions often arise when shaft casings hit strata of saturated sands or silts. In the field, successful stabilization has been achieved in many cases by driving pipes into the ground inside the casing. There appear to be many advantages of using the casing itself as an injection pipe, and this has been investigated in the laboratory using a $4\frac{1}{4}$-in. tube to represent the casing.

Figure 8 is a cross-section of the stabilized soil mass obtained. The outline of the casing is clearly shown, and the shape of the stabilized soil mass is such as to completely withhold water and permit excavation to proceed. Field research is now being done with full scale equipment.

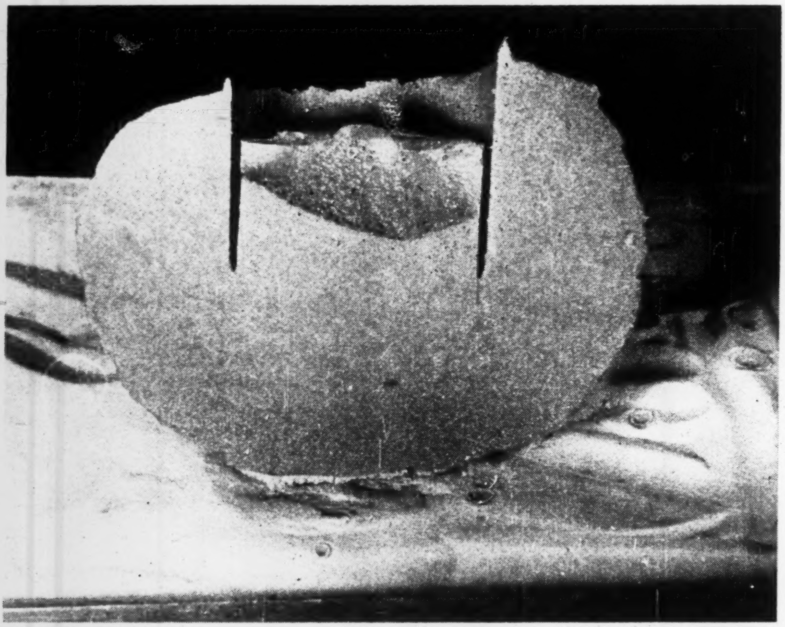

FIG. 8.—Stabilized Soil Mass Showing the Shape Obtained by Using a Casing as an Injection Pipe.

Other laboratory work is still being carried out on various problems connected with stabilized soils. It is still too early to say with complete assurance, but the following results are indicated:

1. The ratio of stabilized soil volume to volume of chemical injected appears to:

(*a*) increase as the injected volume increases,

(*b*) increase as the catalyst concentrations increase (within limits), and

(*c*) increase as the gel time decreases.

2. Stabilized soil will exhibit plastic flow but will not consolidate—that is, water will not be forced out by loading.

3. Alternate freezing and thawing or wetting and drying will deteriorate stabilized soils.

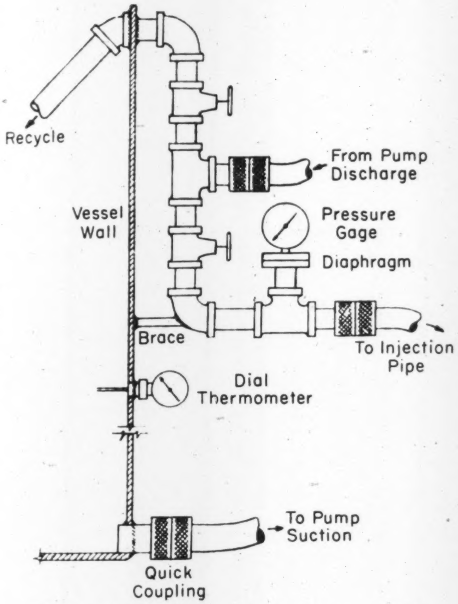
Recycle
From Pump Discharge
Vessel Wall
Pressure Gage
Diaphragm
Brace
To Injection Pipe
Dial Thermometer
To Pump Suction
Quick Coupling

FIG. 9.—Basic Piping System for Injection Equipment.

*Field Work:*

Relatively simple equipment and techniques for placing the chemical in the ground in suitable quantities have been developed. For this purpose the following criteria were established as controlling.

1. Keep the equipment as simple as possible.
2. Use standard equipment, parts and materials.
3. Keep the equipment light enough to be handled and moved manually.

Several sets of equipment have been assembled of varying sizes and capacities. Basically, they all operate through the piping system shown in Fig. 9.

The recycling system is used both for mixing the chemicals and for controlling the injection pressure. The vessel size and the size of the centrifugal pumps will vary depending on the scope of the operation. Weight is kept to a minimum by

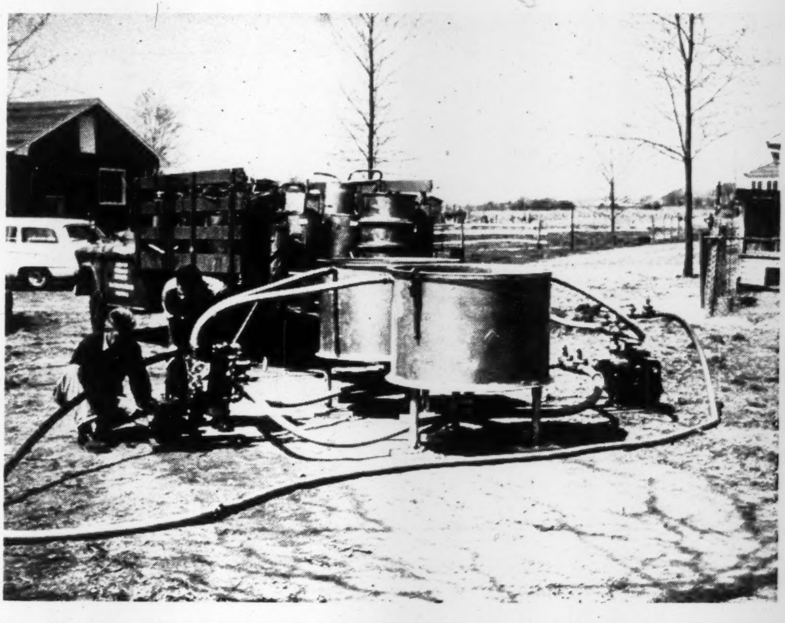

FIG. 10.—Two-Drum Injection System Capable of Continuous Operation at Pumping Rates up to 30 gpm.

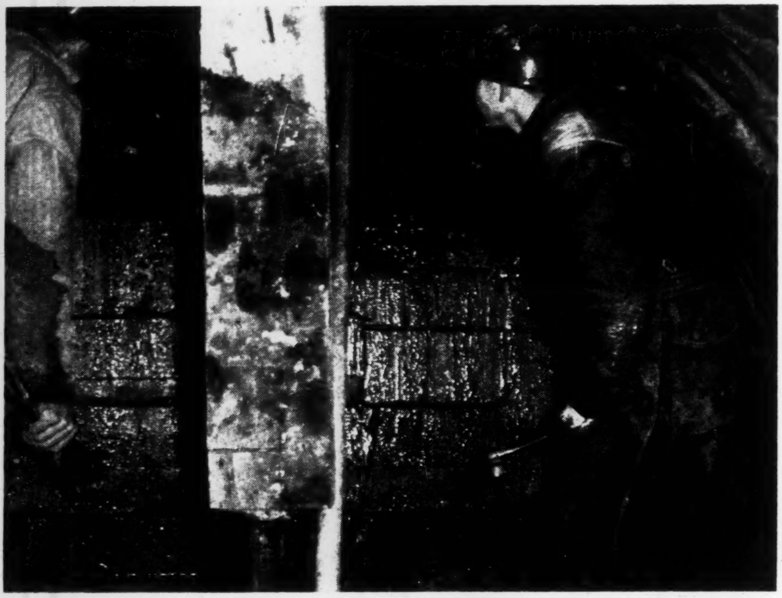

FIG. 11.—Tunnel Face Prior to Stabilization, Showing Oozing Silt and Mud.

use of aluminum wherever possible for vessel and piping.

Figure 10 shows a two-vessel system, by which continuous injections of 20 to 30 gal per min can be maintained. In this setup, mixing is taking place in one drum while the other is being used for injection.

Standard drill rod has been found satisfactory for field work. The lower half of the rod is perforated with 20 or more $\frac{1}{4}$ or $\frac{5}{16}$-in. holes, to facilitate pumping. Drill rod can be driven by sledge or with an air hammer, and will stand considerable abuse.

The one big problem with injection pipes is keeping them clean. Almost all field pumping problems can be traced to the pipes being plugged with soil or gel. A water jet is very adequate for cleaning injection pipes prior to pumping.

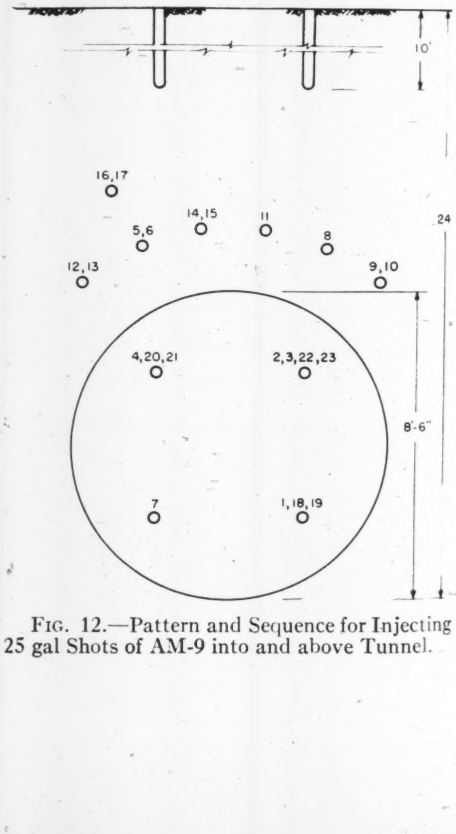

FIG. 12.—Pattern and Sequence for Injecting 25 gal Shots of AM-9 into and above Tunnel.

### TUNNEL STABILIZATION

As part of a new sewer improvement program in Jersey City, N. J., an 8 ft 6 in. diameter tunnel had to be placed for 300 ft under a city street. A shaft was sunk to the invert elevation, about 24 ft below grade, using wood sheet piling. Three external rings of liner plates were erected, then it was attempted to cut out the eye.

Figure 11 shows the tunnel face with two of the top boards removed. There was so much oozing silt and mud, that the face was closed up again, to prevent a possible surface cave in. The source of the water was believed to be an old sewer line, 20 to 30 ft away, and it was thought that after passing this obstacle, no further troubles with running sand would be encountered.

Figure 12 shows an elevation of the tunnel face, indicating the pattern used for placing the AM-9. The circles represent the location of the injection pipes. Both tapered pipe and drill rod were used on this job. The numbers beside the circles represent the sequence in which 25 gal shots of AM-9 were pumped into the soil at the various locations. The numbers up to 12 represent 5 ft long pipes, the higher numbers represent 10 ft long pipes. During one working day, a total of 575 gal of AM-9 were placed as shown.

The following morning, inspection showed that the tunneling crew, which was working two shifts, had removed the bulkhead, placed one complete ring of liner plates, and was excavating for the second. Some small amount of running sand was apparent in the upper left side of the face, and the tunnel crew was asked to stop work for further stabilization. That same afternoon, an additional 14 shots, 350 gal, were placed. Two shots were placed in the upper left quadrant, two in the lower left, and one in the lower right. Four shots were poured into a crevice between the face piling and the ground, and five shots were placed through 10 ft long pipes driven vertically from the surface and leading the tunnel face by about 12 ft.

After this second treatment, the tunnel crew was able to proceed with its work. The tunnel crew foreman estimated that soil conditions were 85 to 90 per cent improved by the chemical grouting operation. Subsequent excavation indicated that in some spots stabilized soil was encountered 30 ft beyond the face.

## DISCUSSION

MR. GEORGE F. SOWERS.[1]—In his paper the author discusses two points—the general makeup of the chemical itself and, second, the field application.

In the chemical itself he discusses the fact the material is, first, water soluble. I think that is a very important point. I have worked with portland cement grouting. This employs a suspension which may actually develop a filter skin in pores of soils and actually strain out the solids so as to produce little penetration of the grout into the soil, and so a true liquid certainly ought to give us a much better penetration than a suspension such as portland cement.

The second thing which I think is important in a mix is a viscosity approaching that of water, because the lower the viscosity (everything else being equal) the greater the penetration into the pores of the soil.

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[1] Consulting Soils Engineer, Law-Barrow-Agee Laboratories, Inc., Soil and Foundation Investigations, Atlanta, Ga.

One point that is not discussed by the author which I have found to lead to some difficulty is the compatibility of these stabilizing materials with the soil itself. First there is the bonding of the stabilizer to the soil. For example, asphalt is a liquid material which can be used as a stabilizer and yet it does not bond to the soil grains very well. As a result, it does not last. So I think this point must be considered, although the author has not discussed it—the bonding of the stabilizing chemical to the soil grains.

A second point which he does not discuss is the effect of the soil itself on the stabilizer. I have worked with some of this chemical which the author furnished to me and found that the highly oxidized red iron soils of the South actually contributed to a reduction in the strength of the stabilizing material that some other soils did not. In other words, there is a reaction between the soil itself and the stabilizing chemical, apparently due to the oxidation of the iron compounds in the soil. And I am also wondering if, in some cases, there might not be a reaction of the stabilizer on the soil. It is conceivable with some chemicals that there may be a reaction that effects some of the clay minerals, adversely, causing them, for example, to swell. I think that is a question that the manufacturer of the stabilizing materials will need to investigate.

The author's discussion of the practical applications is very interesting. I would like to point out one thing. It is not always desirable to restrict the flow of water. I just returned from a job in Tampa, Fla., where every place the contractor restricted the flow of water he created two more leaks that were worse than the first one because of building up of high water pressure. But in the actual application, I think there are two important questions that arise. First, how long does it take for the reaction to take place and how carefully can that be controlled in the field? In a coarse soil with large pores and flowing water, of course, a very short reaction time would be important. But in a fine grain soil where you are after a penetration, and the penetration of course is slow, I think a slow reaction time would be desirable.

The second question is how much does the treatment cost. Of course that is the real question on all the stabilization.

MR. JOHN P. GNAEDINGER.[2]—We have been working with AM-9 since 1952. The first job which we successfully completed was in December 1952, and the job is still holding water. It happened to be a waterproofing job in a basement about 6 ft below the level of Lake Michigan, immediately adjacent to Lake Michigan. We drilled holes through the concrete in the basement wall and floor and pumped chemical into the soil, where it gelled and made the soil impervious. The soil is still impervious and no leaks have recurred. The waterproofing application of AM-9 has been quite successful on more than 100 jobs.

We have also developed several other applications of chemical stabilization, one of the most interesting of which is the prevention of sewer infiltration in Florida. Many sewers in Florida, particularly in the Miami area, are 10 ft or more below the water table, and there is a serious problem of ground water infiltration into the sewers through bad joints. Rather than digging up the joints, which would require the use of well points and considerable excavation, we locate the leaks from manholes and then jet down an injection pipe from the ground surface. Chemical grout is then pumped through the injection pipe. The chemical gels immediately surrounding the leak, with a tendency to flow into the leak. We have been quite successful in this application, stopping most of the leaks with the first injection. Any leaks that do not stop after the first injection are then retreated.

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[2] President, Chemject Corp., Soil Testing Service, Inc., Chicago, Ill.

Some of the sewage treatment plants in Florida are treating more than three times their sewage volume, due to infiltration, and prevention of such infiltration by chemical grouting pays for itself in economies within one year in many cases.

Another application which we have just finished was the use of chemical grouting to permit excavation into an open cut along Lake Michigan, where a trench had to be dug about 10 ft below lake level, located about 5 ft from the lake. We pumped in chemical to solidify the sand. They have opened the excavation and found it almost completely dry, avoiding the need of expensive well points and presenting a much easier construction problem.

We are also using chemical stabilization to permit caisson excavation through sandy soils below the water table. More information on this process will be presented at a later date.

In answer to a statement that was made by the previous discusser, it is my opinion that chemical stabilization with AM-9 is economical for many purposes, and in many cases it is cheaper than cement grouting or sodium silicate grouting even though the materials used in our chemical grouting are much more expensive. The principal advantage is in the ease of injection due to the low viscosity of the AM-9 grout, and the easy control over the rate of gelation. Furthermore, with chemical grouting only one solution need be pumped in, in contrast to the sodium silicate grouting which generally required two solutions.

Mr. Gordon A. Fletcher.[3]—My first question is for clarification.

The literature I have read on this subject in the past refers to this chemical as AM-955. The author referred to it as AM-9. I would like to know if there is any difference between those two.

My second question has to do with the soil waters which the chemical is calculated to block off or solidify. My question has to do with the chemical nature of such soil waters. Are there any such chemicals normally found in soils which would prevent the polymerization of this gel?

My third question has to do with the matter of the pressures required to force the chemical solution far enough radially to be effective and whether those pressures, if the horizon is at a great depth below the hydrostatic head, if those pressures are high enough to prevent, or if they actually help, polymerization.

Mr. Eugene Matyas.[4]—I have one short question to ask of the author. He states that to the best of present knowledge the final gel is not toxic except that it may contain small amounts of unreacted AM-9. I would like to ask if any toxic solution might leach out of the final gel; and if so, can chemical grouting with AM-9 be used in the vicinity of a potable water supply without creating a health hazard?

Mr. Karol (*author's closure*).—Laboratory work has shown that the presence of ferrous or cuprous ions tends to reduce the induction period and gel strength of AM-9 (formerly AM-955). This condition is rather severe with the catalysts which had been in use at the time AM-9 samples were sent to Mr. Sowers. With an AP-NTP system, the effects of these ions can be overcome by increasing the catalyst concentration and using an inhibitor to control gel time. With an AP-DAP system, the effects of ferrous and cuprous ions are negligible. Both iron and copper ores have been gelled successfully in the laboratory, and field work in exploration hole drilling has been successfully carried out in Canadian iron mines. In this instance, the sidewalls of the drill holes were stabilized to permit drilling without casing.

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[3] Assistant Vice-president, Raymond Concrete Pile Co., Gow Division, Borings and Soil Investigations, New York, N. Y.

[4] Civil Engineer, Design Division, Public Works, Philadelphia Naval Shipyard, Philadelphia, Pa.

To the best of our knowledge, there are no chemicals normally present in soils that would prevent gelation from occurring. We have experienced several isolated instances in which soils with a low pH inhibit the AM-9 solution. That is, they increase the normal induction period. This can be overcome, however, by adjustment of the catalyst proportions.

In regard to bonding, we believe that any chemical bonds that are formed with the soil grains are weak, probably produced through Van der Waals' forces or hydrogen bonds. Mechanical bonding does occur as evidenced by the unconfined compression strength of gelled sands, which would normally have no strength. Field work has verified this, too. Gel placed in rock fissures has withstood water heads exceeding 100 ft.

In the laboratory, induction periods can be controlled and reproduced very accurately. Since soil and ground water may have minor effects, we have made it a standard practice to run field tests at the site to verify gel times. If this is done, field control of induction period should be very good (within 5 per cent) for gel times less than an hour and within 10 per cent for longer gel times.

All of our field work has been done at the lowest pressures consistent with placing the desired quantity of solution in a reasonable time. For shallow work (within 50 ft of the ground surface) most applications have been made at 50 psi or less. For deep hole drilling and for work in mines and deep shafts it has sometimes been necessary to use 200 to 300 psi to place the solution. To the best of our knowledge, pumping pressure has no effect on induction period or gel strength.

In regard to toxicity, it is certainly possible for unreacted AM-9 to be leached out of a solid gel. However, we believe the amount of unreacted AM-9 to be very small, and consequently the possible concentration of AM-9 in a potable water supply will be very small. In recent tests AM-9 was added to a fish tank in amounts to give a million to one dilution. Although this treatment was repeated every week for many weeks, there were no apparent ill affects on either fish or plant life. At this time, we certainly do not rule out work near a potable water supply. However, each application must be very carefully analyzed if potable or recreational water is nearby.

In regard to cost of chemicals, most of our work has been done with 10 per cent solutions. At this concentration, the cost per gallon, including AM-9 and the necessary catalysts is about 75 cents.

# DESIGN AND DEFLECTION CONTROL OF BURIED STEEL PIPE SUPPORTING EARTH LOADS AND LIVE LOADS*

By Russell E. Barnard[1]

## Synopsis

This paper deals only with round, or nearly round, smooth, unreinforced steel pipe such as water pipe buried under fills or in trenches carrying the earth cover and live load if any. The basic principles, however, apply equally well, with proper regard for inherent strength, to other pipe used as culverts or drainage structures under highways and railroads, or through dams.

A round flexible steel pipe when buried tends to lessen its vertical diameter and increase its horizontal diameter under the weight of the earth cover and the live loads, if any. The outward thrust of the pipe places a compressive load on the soil at the pipe sides. Calculations of vertical settlement of structures have been made for many years in accordance with certain established principles. In this paper these same principles are used to calculate side-wise settlement or outward movement of the pipe wall against the soil. The horizontal movement is related to the vertical deflection which can then be calculated for a given set of conditions. Also, the amount of vertical ellipsing, strutting or stulling necessary to produce the desired horizontal dimension can be determined.

Calculated deflections are compared with measured deflections for several average and several extreme cases. These comparisons show an interesting degree of accuracy over a wide range of conditions.

Design data are given for earth load and live load intensity, the strength of smooth steel shells under such loads, and the engineering properties of several typical remolded soils which may surround the pipe and act with it in a composite manner. Formulas and tables provide simple means of calculating pipe wall thickness and determining vertical diameter shortening, or regulating it, to be compatible with the earth envelope properties achieved through different degrees of soil compaction around the pipe.

Three basic factors are involved in engineering structures: (1) the design, (2) the material, and (3) the environment. All three must be evaluated. Designwise there are determinate and indeterminate calculation problems. To aid in designing indeterminate structures soundly and economically, engineers have found no substitute for satisfactory experience. Simulated service tests are now supplementing experience.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

[1] Advisory Engineer, Armco Drainage and Metal Products, Inc., Middletown, Ohio.

Rapid progress is being achieved through designing for safe load rather than stress in all cases (**1**,**2**).[2]

The laboratory tests made by mechanical testing men provide the designer with the measured strength and ductility of steel. Tests made by soil mechanics men are measuring the engineering properties of soils. The data from these tests enable the designer to make an analysis of the environment of underground steel conduits in a manner not possible before.

## THE PIPE AND ITS ACTION

When there is little or no side support, a steel pipe underground functions as an elastic ring loaded at top and bottom. Relatively thick rings carry more load with less deflection than do thinner rings and may be said to possess greater "ring strength." However, in a given case, if the ring strength is not sufficient to carry the full load, the pipe deflects and soil side support builds up to carry that part of the load not carried by the ring. Considering progressively pipes having less and less "ring strength," it is seen that the resisting action in the different walls must change from that of a ring loaded at top and bottom to the arch action of a totally enclosed ring. In the latter case, the confined ring must possess sufficient "arch strength" to adequately resist the load even though its ring strength may be nil. Here such a ring is termed flexible.

Experience with flexible steel pipe underground has shown that bending moment resistance to external load is relatively unimportant except where there are high live loads and shallow cover; that the earth envelope is as much a part of a buried steel pipe structure as is the concrete in a steel reinforced-concrete pipe; that soil compac-

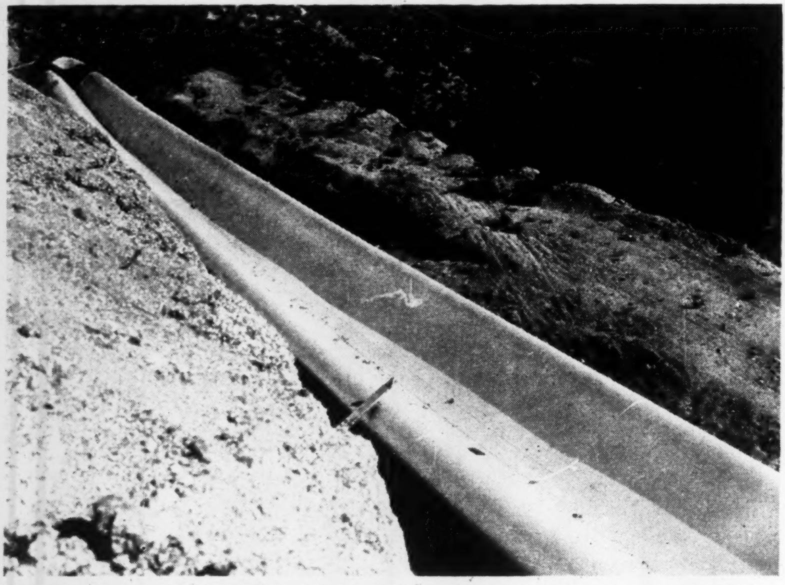

FIG. 1.—Bouquet Canyon Pipe Line Flattened in 1936 by Failure of Vacuum Relief Valve.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1255.

tion methods used in years past have resulted in satisfactory installations and that calculating the pipe wall thickness using the membrane theory (3,4) is good practice for the wall thickness-diameter ratio of steel pipe ordinarily used. The membrane theory neglects bending stresses in thin shells on the same basis that bending stresses are ignored in wire and rod reinforcing wound around pipe or made into reinforcing cages.

Satisfactory performances of steel pipe for over a century have proven that the principal function of a structure is to resist loads and that apparent bending stresses based on elastic theory are not of importance in themselves when the ductility of the material in the shell permits deformation without service failure. A rather unusual instance of such deformation is shown in Figs. 1 and 2. Ordinary demonstrations are given by ordinary water pipe. A rule-of-thumb has been used for over 50 years to fix the minimum practical wall thickness for steel water pipe at $\frac{1}{2}$ per cent of its diameter. Large footages of pipe 48 in. in diameter with $\frac{1}{4}$-in. wall, conforming to this rule, have been used for many years at usual pipe depths without any collapse failures. In fact, there are many satisfactory installations of steel water pipe having thickness less than called for by this rule (5). Such thin pipes cannot possibly resist earth pressures through any ability to develop adequate bending resistance in the pipe wall. It will be shown that nevertheless they have large safety factors in compressive arch action for the loads they carry.

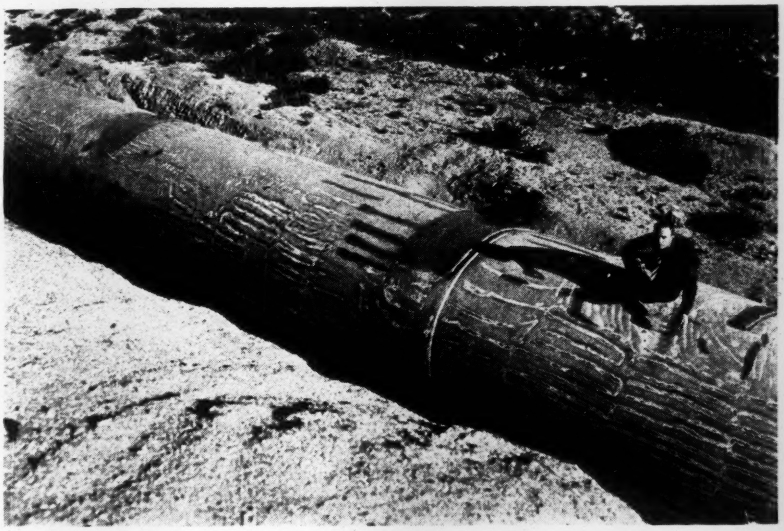


FIG. 2.—Bouquet Canyon Pipe Line Restored to Form by Water Pressure and Still in Service in 1957.

A typical stress-strain diagram for the steel here under discussion is shown in Fig. 3. Steels of this kind made to ASTM specifications have specified minimum yields on the order of 27,000 to 35,000 psi; ultimate strengths 48,000 to 60,000 psi and elongation in 2 in. of 20 to 30 per cent.

### *Definition of Failure:*

When deformation is mentioned, the engineer accustomed to thinking in terms of flexure or bending-moment formulas in rigid construction may contend that permanent deflection can only occur after the yield point has been passed and that therefore a pipe so stressed has failed structurally and is dangerous. This is true of brittle materials but experience proves otherwise for ductile materials. The simplest proof lies in recognizing that the steel in a finished pipe has, in the manufacturing process, been cold coiled, uncoiled, bent, curved or twisted a number of times and has been stressed beyond the yield point each time. Despite this, after all these operations have been completed, the finished steel pipes or pressure vessels are used for all manner of high pressure work without fear or hesitation.

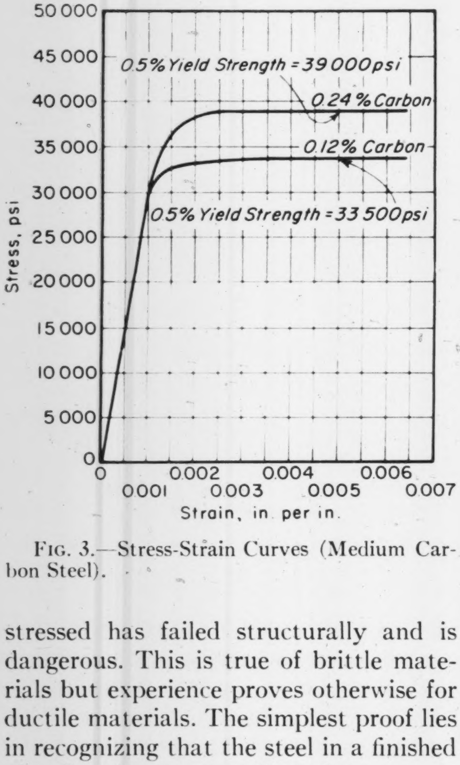


FIG. 3.—Stress-Strain Curves (Medium Carbon Steel).

If the engineer still is hesitant to stress a part of the finished pipe wall beyond the yield point again by slightly deflecting it underground, let him consider what happens to the test specimen by which the pipe strength is measured according to specification. In most cases it is sliced as a ring from the end of a finished pipe, cut at one side, uncurled from the circle into a flat piece, and then put in a tension testing machine which proceeds to show that after once more passing the elastic limit, the steel still possesses the specified strength. In a way, the deflection underground is simply a finishing forming operation.

Therefore, where steel pipe such as is here discussed is concerned, the word failure must define a state of falling short in satisfactory performance and not a state in which localized stresses appear to pass the yield point of the material as judged by the results of bending-moment formula analysis.

## ARCH STRENGTH

That a confined thin steel shell under outside load must act in compression with bending resistance relatively unimportant can be demonstrated by laboratory test and by the manufacturing procedure of forming steel pipe in a die.

### *Proof by Laboratory Test:*

An endeavor was made to demonstrate photoelastically that the postulation of load distribution around embedded steel pipe used in this paper was not seriously in error. A hole was drilled in a $\frac{3}{8}$-in. thick plastic plate to accommodate an aluminum ring 1.25 in. in diameter 0.034 in. thick, and $\frac{1}{2}$ in. long. A slot $\frac{5}{8}$ in. wide extended from the hole to the top of the plate. A steel loading plunger $\frac{5}{8}$ in. wide operated in this slot. The contact face of the plunger was ground to fit on the ring. It delivered a load covering one-half the tube diameter.

*First Series of Tests.*—In the first series of tests the hole size was a slip-fit for the ring. This would come as near developing uniform radial pressure in the ring as could be expected considering the elements and factors involved in the test. Figure 4 shows the fringe pattern. It indicates a relatively uniform radial stress except near the area of application of a concentrated load where a different pattern would naturally be expected.

*Second Series of Tests.*—In the second series of tests the hole was made $\frac{1}{32}$ in. oversize of the ring. Thus the ring was allowed to deflect about $2\frac{1}{2}$ per cent vertically before full contact was established in the horizontal direction. The idea here was to develop a pattern at the extremities of the horizontal diameter which would resemble the familiar onion-like bulb of pressure existing under footings delivering load to soil. Figure 5 shows the stress pattern achieved. To be noted are the beginnings of the "ears" which when full-grown are postulated as being similar to that bulb-of-pressure under a footing load.

*Stresses in Laboratory Ring Tests:*

Noteworthy are the calculated stresses in the model ring. As an elastic ring without side support, placed between the heads of the testing machine, it held 30 lb with a deflection of about $2\frac{1}{2}$ per cent. The maximum load in the first series of tests was 200 lb. In the second series the maximum was 250 lb. The stress determined using the method for arch-load (see further for definition) is 5900 psi for the 200-lb load and 7300 psi for the 250-lb load.

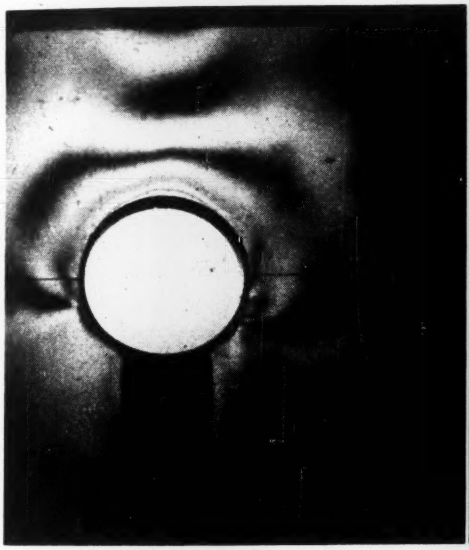


FIG. 4.—Fringe Pattern Around Ring in Slip-fit Hole.

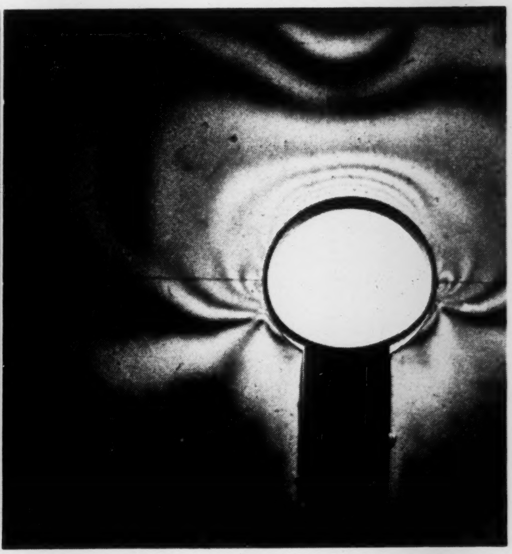


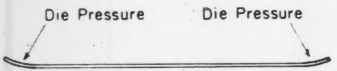
FIG. 5.—Fringe Pattern Around Ring in Oversize Hole.

Calculated on the basis of section modulus resistance, the apparent stress in the ring under the plunger load is about 3,750,000 psi for the 200-lb load and 4,700,000 psi for the 250-lb load.

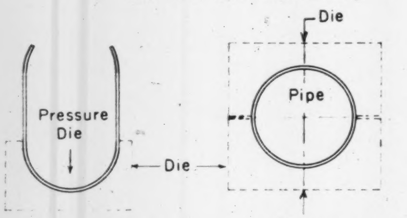
The material in the ring had a yield strength of about 55,000 psi and an ultimate of about 75,000 psi. The ring used was not harmed. After being removed from the testing machine, it would roll on plate glass without showing a balance point.

*Proof by Die Forming Pipe:*

The process of manufacturing high-test line pipe furnishes continuous test proof of the tremendous arch loads which can be carried by smooth wall steel pipe. The forming operations are shown in Fig. 6. The pipe is made from steel plates having a length equal to the finished pipe section and a width somewhat greater than the pipe circumference. The first operation consists of die-forming the sides of the plate to a radius approximating that of the finished pipe as shown in Fig. 6(*a*). The second operation is performed in a U-shaped die with the result shown in Fig. 6(*b*). In the last operation as shown in Fig. 6(*c*), two semi-circular die sections are brought against the U-shaped pipe, forcing it into a complete circle with sufficient pressure between the plate edges to permit electric-resistance flash welding. The final closing of the die forces weld metal from the welded joint inward toward the center of the pipe and outward at the top. Such operations are performed on 30 or 36-in. pipe having a wall thickness as little as $\frac{1}{4}$ in. It is necessary to recognize the important part the engineering properties of the pipe material and the die material have in this manufacturing operation.



(a) First Operation Forming



(b) Second Operation Forming (c) Third Operation Closing

FIG. 6.—Forming High Pressure Steel Pipe.

## GENERAL THEORY

Almost all analyses of underground conduits to date have been directed at the structure itself and the load it carries. Such attention as has been given the soil surrounding flexible conduits has been confined to "tamping" or "not tamping" or to assigning values to a single earth assistance selective constant (6). Such an evaluation of earth assistance does not account for the fact that soil pressure tests show that the load-pressure relationship is not linear, and that soils do not react to load in an elastic manner within the range of soil pressures ordinarily found in common pipe installations.

A rigorous analysis of the problem of underground conduits is probably impractical, at least at this time. There are unmeasured variables in field installation, in backfilling and compacting and in precise knowledge of soil engineering characteristics. Under such circumstances extreme accuracy is not expected. The semi-empirical approach is, however, justified as a practical approximation.

The soil which surrounds a pipe in a trench or in a fill is a remolded soil. Assume that it is required to determine the settlement under load *P*, Fig. 7, in a fill made of two kinds of materials and built on a natural ground at plane 3. There are several ways to do this. One method is to make properly controlled triaxial tests of the soils involved to determine their engineering properties (7). This method is used in this paper for illustrative purposes because the data on several typical remolded soils were available (8). Settlement may be determined by void ratio - pressure data on soil samples or, in more approximate form, by using data developed by Proctor (9). This latter subject will be discussed later.

In Fig. 7, the load distribution in the soil due to load *P* is found by methods of Boussinesq (10) and a "fade-out" point is established for calculation purposes. When triaxial test specimen A is tested with confining pressure equivalent to the height of earth column $z_1$, and axial pressure reaching the specimen location, the ratio of its shortening $s_A$ to its length $l$ may be considered the same as the ratio of settlement $S_1$ to thickness of layer $L_1$ which it represents. If $s_A$ is expressed as a percentage of $l$, then $S_1$ is the same percentage of $L_1$.

The same reasoning applies to specimens B and C and layers $L_2$ and $L_3$. The sum of all the settlements equals the settlement of the load $P$. The accuracy of the findings is influenced by the many uncertainties of sampling, testing, and the calculations of underground load distribution.

The foregoing calculation is an estimate of initial settlement. Settlement due to time-consolidation is additive. In this paper, time-consolidation is accounted for by a lag factor and is discussed later.

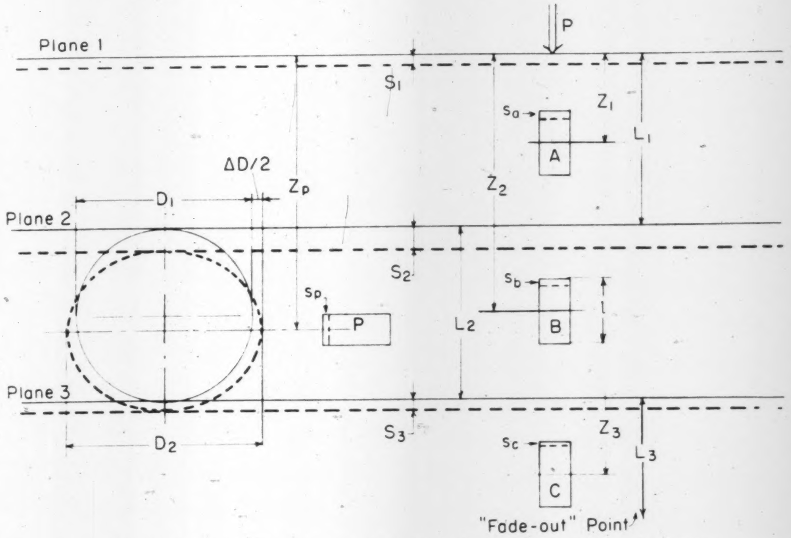


FIG. 7.—Illustrating Settlement of Fill and Deflection of Flexible Pipe.

The left-hand portion of Fig. 7 indicates a steel pipe as it might be installed in a fill such as considered above. The pipe is shown as having settled with plane 3 and deflected enough to occupy the space between planes 2 and 3 after initial settlement. For purposes of

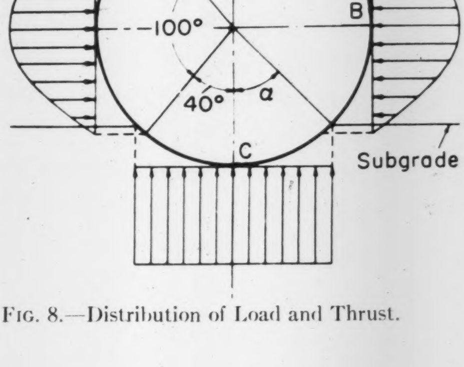


FIG. 8.—Distribution of Load and Thrust.

engineering design, it is postulated that the pipe is supporting the full weight of the prism of earth above it because there are no friction planes to modify the load. For a flexible pipe this is the worst situation and can be used for calculation purposes.

The horizontal thrust of the pipe side against the soil is assumed to be distributed in some such fashion as is indicated in Fig. 8. Since the total of all forces must be in equilibrium, the force of maximum intensity on the horizontal diameter must exceed the intensity on the vertical diameter by some amount. It is affected also by the instantaneous radius of the pipe in contact with the soil at a given point. This is discussed under "intensity of contact pressure."

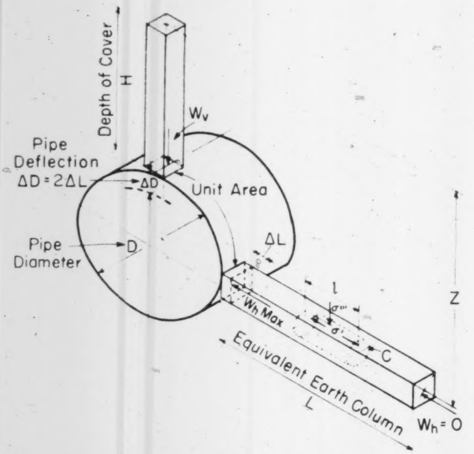


FIG. 9.—Illustrating Equivalent Earth Column.

The size of a footing is a factor in its settlement and therefore the diameter of a pipe is a factor in its "side-wise" settlement. The greater the size or diameter, the greater the settlement for given load intensity. The settlement or passive deformation at the pipe side may be expressed in terms of the pipe diameter.

The loading at the side of a pipe is similar to that for a flexible strip of infinite length uniformly loaded.

The passive deformation for a given remolded soil with given compaction may be determined from triaxial test data for that soil corresponding to a location $P$ on the horizontal pipe axis. Of course, the deformation also may be determined by other methods if available. For a given soil, the load to cause a given deformation becomes greater as the depth of the soil below the surface becomes greater. This is important in determining pipe deflection.

The basic concept of the equivalent earth column method of calculation is shown in Fig. 9. The engineering properties of the soil are determined for a position on the horizontal pipe axis.

## DEFINITION OF TERMS

Further discussion will be facilitated by defining and using certain terms.

*Ring-load.*—The load carried by the conduit shell as determined by moment-modulus calculations for ring diameter changes of about 5 per cent or less.

*Ring-aid.*—The horizontal thrust toward the pipe due to active earth pressure. May be calculated on the basis of $\frac{1}{3}$ vertical pressure for sand and $\frac{1}{2}$ for clay. Used only to reduce ring-load, not arch-load when the internal water pressure is great enough to round the pipe underground after installation and create a void between pipe side and backfill.

*Arch-load.*—The load carried by the conduit shell under external radially acting pressure determined by using the Barlow formula:

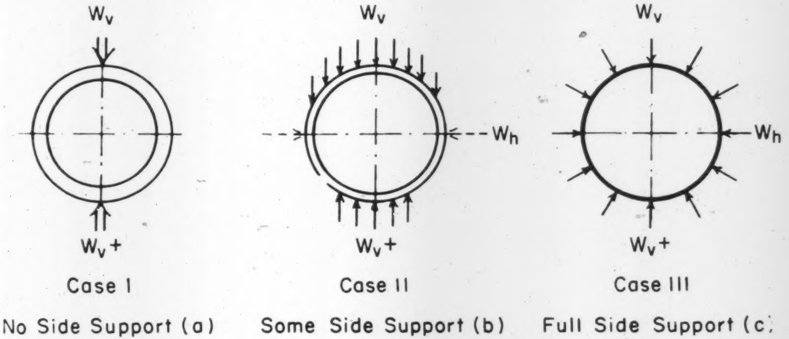
$$2St = pD \quad \text{.............} (1)$$

$$\text{or} \quad St = pR \quad \text{.............} (2)$$

where:

$S$ = stress, psi,
$t$ = wall thickness, in.,

$p$ = pressure due to height of cover above pipe, psi,
$D$ = conduit diameter, in., and
$R$ = instantaneous radius of deformed conduit at given point, in.



No Side Support (a) Some Side Support (b) Full Side Support (c)

FIG. 10.—Types of Conduits and Loading.

The indicated stress in the pipe wall from Eqs. 1 and 2 is, respectively:

$$S = \frac{pD}{2t} \quad \text{. . . . . . . . . . . . . . (3)}$$

or

$$S = \frac{pR}{t} \quad \text{. . . . . . . . . . . . . . (4)}$$

*Transfer load.*—The load in excess of the ring-load which is imparted to the soil at the sides of the pipe.

*Passive deformation.*—The horizontal "settlement" or distance the soil is compressed by the transfer load.

*Contact pressure.*—The measure of the force acting between metal surface and soil surface in an approximate horizontal direction.

## RING STRENGTH AND ARCH STRENGTH

Three different cases of pipe resisting action are recognized:

*Case I*, Fig. 10(*a*).—In this case, the wall thickness and diameter of the pipe selected to meet pressure and other service requirements is such that its ring strength can carry satisfactorily all earth loads upon it without undue deflection. This case is quite frequent in water works practice and no problem occurs in installation. Ring strength for 2 per cent deflection is given in Fig. 13.

*Case II*, Fig. 10(*b*).—Here the pipe ring strength is sufficient to carry part of the earth load and live load but not all of it

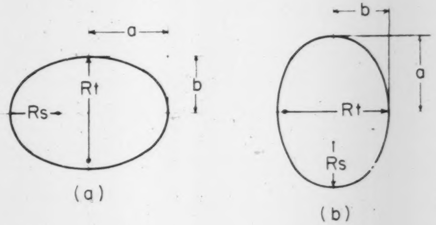


FIG. 11.—Instantaneous Radii of Round Pipe Expanding Horizontally (*a*) and Ellipsed for Installation (*b*).

TABLE I.—VALUES OF SHAPE FACTOR, *F*.

| Pipe Deflection, per cent of $D$ | Values of $F$ for Pipe Elongated as Shown | |
|---|---|---|
| | Deflected Horizontally, Fig. 11 (*a*) | Strutted Vertically, Fig. 11(*b*) |
| 0.0 | 1.25 | 1.25 |
| 0.5 | 1.29 | 1.21 |
| 1.0 | 1.33 | 1.18 |
| 1.5 | 1.37 | 1.14 |
| 2.0 | 1.40 | 1.11 |
| 2.5 | 1.45 | 1.08 |
| 3.0 | 1.50 | 1.04 |
| 3.5 | 1.55 | 1.01 |
| 4.0 | 1.60 | 0.98 |
| 4.5 | 1.65 | 0.95 |
| 5.0 | 1.70 | 0.93 |

without undue deflection. Some side support must be afforded by the earth.

*Case III*, Fig. 10(*c*).—Here the pipe is a completely flexible thin ring and by itself can carry very little, if any, of the live and dead loads without undue deflection. Full mobilization of earth assistance is needed to retain the degree of roundness required. The enveloping forces are essentially radially acting. The ring is a confined arch under compression. It will continue to act as an arch as long as the curvature remains convex against the earth. Arch load creating compressive stress of 7500 psi is given in Fig. 14.

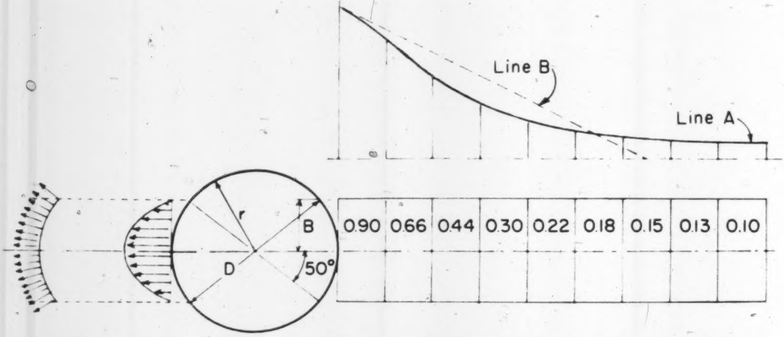


FIG. 12.—Method of Determining Length of Equivalent Earth Column.

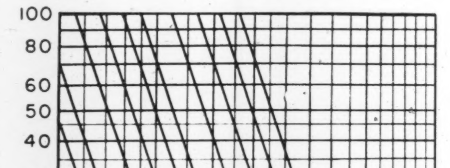


FIG. 13.—Theoretical Height of Cover Ring Load for Steel Pipe with 2 per cent Deflection.

## NOMENCLATURE

Referring to Fig. 9, let:

$D$ = pipe diameter, in.
$\Delta D$ = vertical deflection of pipe, in. (decimal of diameter),
$w_v$ = total vertical unit load on pipe, psi,
$w_{vd}$ = unit dead load on pipe, psi,
$w_{vl}$ = unit live load on pipe, psi,
$F$ = shape factor (a selective constant),
$w_p$ = unit load, ring load carried by pipe when $\Delta D = 0.02D$ (values from Fig. 13), psi,
$w_a$ = unit load, ring-aid afforded by active soil pressure, psi,
$w_h$ = unit horizontal load transfer-load contact pressure, psi,
$W$ = total live load plus dead load, psi,
$L$ = effective length of equivalent earth column, in. (or in terms of $D$),
$\Delta L$ = passive deformation, in. (or decimal of $L$),
$\epsilon$ = strain in soil, in. per in. for decimal of tested length, under load of intensity $w_h$
$\epsilon_{w_h}$ = strain (per cent) corresponding to given $w_h$ in Figs. 15 to 21 inclusive.

### INTENSITY OF VERTICAL LOAD—$w_v$

Total vertical load equals dead load for height of cover $H$ plus live load, impact, etc:

$$w_v = w_{vd} + w_{vl} \qquad (5)$$

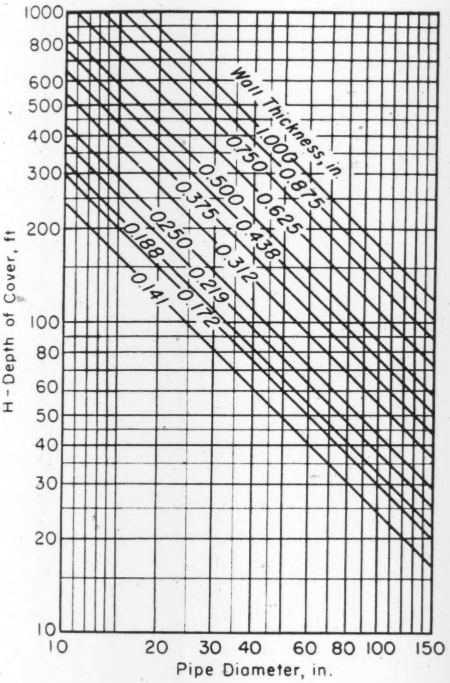


FIG. 14.—Theoretical Height of Cover Arch Load for Steel Pipe with Full Side Support.

When a trench is backfilled by ordinary methods and without tamping, some portion of the active pressure of the soil in the backfill acts against the side of the pipe and tends to support it. For this case

$$w_v < w_p + w_a$$

and

$$w_v - w_a < w_p \qquad (6)$$

If $w_p$ is not greater than $w_v - w_a$, Eq 6 should be ignored altogether.

### INTENSITY OF CONTACT PRESSURE

Intensity of contact pressure between pipe and soil is dependent (1) on the vertical load, (2) on the degree of stiffness of pipe as defined under "ring strength and arch strength," and (3): on the instantaneous radius of the point of contact considered—in this case the extremity of the horizontal axis.

A flexible steel ring or pipe without appreciable ring strength or stiffness (Case III) is best thought of as being a stress gage itself when confined by an earth envelope and supporting earth loads. If the pipe is held perfectly round there is no bending moment and the compressive stress in the wall is given by

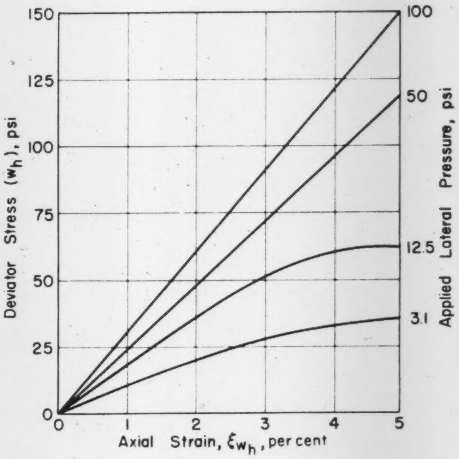


FIG. 15.—Typical Material—Sand-Well Graded—Test 1-T.

$$S = \frac{pR}{t} \qquad (7)$$

It is obvious that this same stress must prevail in every portion of the pipe periphery. The vertical load initiates the stress. The confining pressure in the soil must maintain that same stress at every point or the pipe will change shape.

Therefore, in a truly round pipe the confining pressure must act radially and be equal to $p$ in Eq 7.

Also, it is seen in Eq 7 that for a given stress and thickness, the value of $p$ must increase if $R$ decreases and decrease if $R$ increases. Therefore, to maintain this circumferential compressive force equally at all points in the steel after a round pipe has deflected, the outside radially acting confining force, or earth pressure, must rise at points of lesser radii.

In an initially round pipe of diameter $D$, $a = b$ where $a$ and $b$ are as shown in Fig. 11. As the pipe deflects, Fig. 11($a$), the amount by which $b$ is lessened is added to $a$. The unit vertical load on a flattened pipe creates a tangential compression stress in the wall at the vertical diameter given by Eq 7 with $R$ equal to the instantaneous radius which occurs at the vertical axis of an assumed ellipse. This top and bottom radius is larger for the flattened pipe than for the original round pipe and creates a higher stress in the pipe wall. Also, the instantaneous radius at the extremity of the horizontal axis is smaller for the flattened pipe than for the original round pipe and, since the same stress in the wall must be maintained there to prevent pipe wall movement, the contact pressure on the horizontal axis must be greater than that on the vertical axis. The ellipsed pipe shown in Fig. 11($b$) is discussed later.

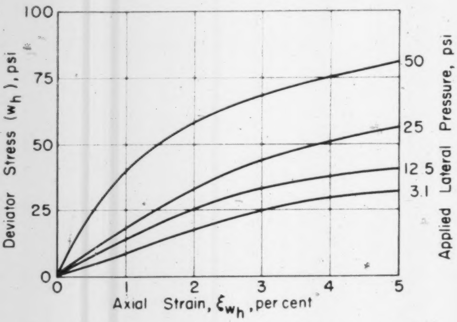


FIG. 16.—Typical Material—Sand with Clay Binder—Test 10-T.

## SHAPE FACTOR

The calculation constant which accounts for this condition when combined with the figure for length of equivalent earth column is here termed the "shape factor."

For example, consider the case of a round pipe of radius $r$ deflected 2 per cent of its diameter. In Fig. 11($a$) $b = (1.00 - 0.02)r = 0.98r$; $a = (1.00 + 0.02)r = 1.02r$. The instantaneous radius $R_T = \frac{a^2}{b}$; the instantaneous radius $R_s = \frac{b^2}{a}$ and $\frac{R_T}{R_s} = \frac{a^3}{b^3} = 1.127$. Multiplying 1.127 by the constant 1.25 in Eq 11 gives 1.40 which is shown in Table I and is the value of $F$ in Eqs 8 and 11. Table I gives values of $F$ for other deflections of flattened pipe and also values for ellipsed pipe shown in Fig. 11($b$). Considering the relative accuracy of other factors, hair-splitting the values of $F$ appears unjustified for deflections of 2 per cent and less used for water pipe design. The value of $F = 1.35$ is used in the calculations for round pipe deflecting 1 to 2 per cent in this paper; fits expe-

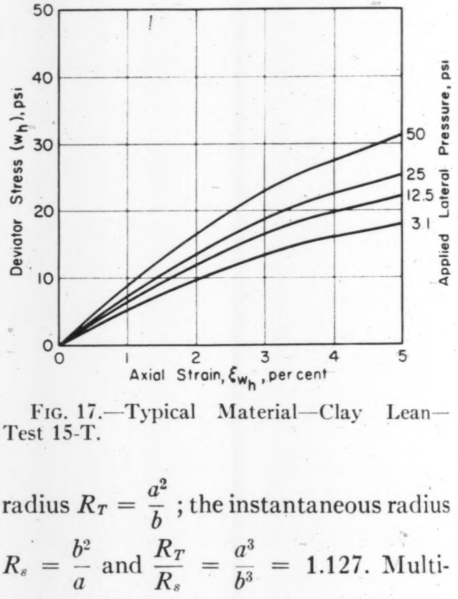


FIG. 17.—Typical Material—Clay Lean—Test 15-T.

rience very well and applies equally well to pipe which is lined with cement or with coal tar enamel. Noteworthy is the reduction in the value of $F$ and the intensity of contact pressure due to ellipsing the pipe.

The intensity of contact pressure covering cases I, II and III can now be stated as:

$$w_h = F(w_v - w_p) \qquad (8)$$

## LENGTH OF EQUIVALENT EARTH COLUMN

First, a "footing width" must be assumed. This "width" is taken as a chord subtended by a central angle of 100 deg as shown in Fig. 12 (see also Fig. 8). The dimension $B$ is the usual one-half footing width used in determining vertical stress at selected depths below that footing. In Fig. 12, line $A$ represents the influence line for calculating vertical stress under a strip when the length to width ratio lies between 2 and infinity as shown by Terzaghi (10). The stress central in each increment of $B$ is shown in the corresponding blocks. Ten per cent of stress is yet unaccounted for in the ninth block from the soil-pipe contact line.

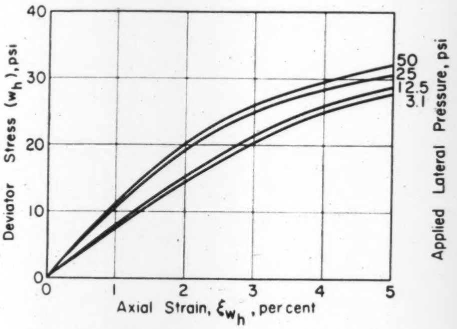


FIG. 18.—Typical Material—Clay—Very Plastic—Test 20-T.

Line $B$ represents the hypotenuse of a right angle triangle which contains the same area (between the line and the horizontal axis) as that under line $A$, except that the missing 10 per cent stress has been added. On this basis, the horizontal leg of the triangle has a length of approximately 6.5 times $B$. This distance from the pipe face is therefore considered the point of practical fade-out for calculation purposes. Therefore, in Fig. 12:

$$B = r \sin 50 \text{ deg} = 0.766\, r$$

Or in terms of pipe diameter $D$,

$$B = 0.383\, D$$

Then the length $L$ of the equivalent earth column is:

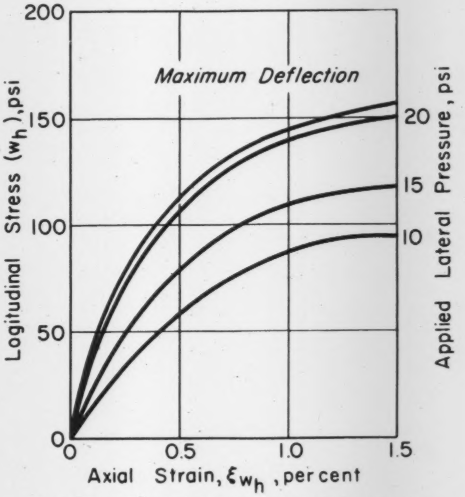


FIG. 19.—Graded Gravel at Maximum Density.

$$L = 6.5\ (0.383)\ D$$
$$L = 2.49\ D \quad \text{or say} \quad L = 2.5\ D \qquad (9)$$

## PASSIVE DEFORMATION

On the basis that the deformation or shortening of the equivalent earth column under the fading load is equal to that produced by the average load over full column length:

$$\Delta L = \frac{w_h}{2}\,(2.5D)\epsilon$$

$$\Delta L = 1.25 w_h D\epsilon \qquad (10)$$

The shape factor $F$ was mentioned previously. When substituted in Eq 10:

$$\Delta L = F w_h D \epsilon \quad \text{.........} \quad (11)$$

The values of $F$, for use in Eqs 8 and 11, are given in Table I for round pipe which has been decreased in vertical diameter and also for pipe which has been increased or ellipsed in vertical diameter for construction purposes. For pipe normally installed round and allowed to deflect vertically under load, the horizontal thrust increases as the deflection increases.

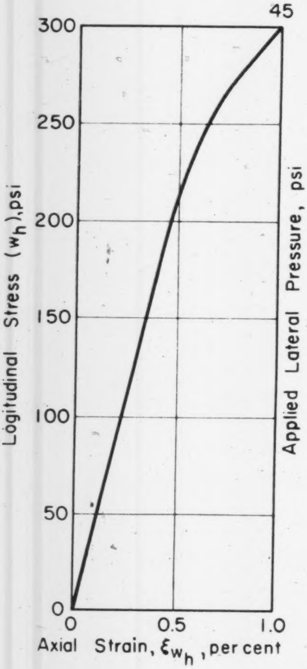


FIG. 20.—Crushed Slag at Maximum Density.

When pipe is formed, strutted or stulled to increase its vertical diameter and it remains that way after installation, the horizontal thrust decreases as the amount of strutting increases. However, this ellipsing cannot be carried too far, or the active pressure at the pipe side may reverse the convexity of the wall. Equation 4 may be used to investigate this latter condition. The ring strength must be considered.

An important factor often overlooked in the case of ellipsed pipe is that the area of an ellipse is less than that of a circle with the same circumference. Therefore, the vertical load causing flattening of an ellipsed flexible pipe tends to actually make the pipe occupy a larger space in the soil as it expands horizontally and tends to become round. This action causes a consolidation of the earth envelope and builds up pressure by reducing the void ratio until the pipe becomes round. An initially round pipe deflecting vertically actually occupies less and less volume as the deflection increases and must develop side support without the special aid afforded the ellipsed pipe.

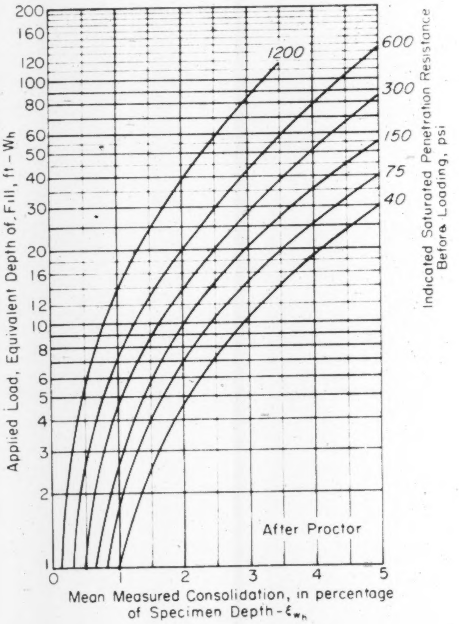


FIG. 21.—Showing Percentage Consolidation at Different Depths of Fill for Soils with Given Proctor Indicated Saturated Penetration Resistance Before Loading.

## TIME-LAG FACTOR

The passive deformation determined using Eq 11 is the *initial* "side-wise settlement". As with vertical settlements, experience shows that the side-wise settlements increase slowly with time. The increase becomes slower and slower, however, until after a period of years virtual stability is attained. In a particular case when engineering properties of a given soil are known, the time-consolidation rate can be determined. Initial and subsequent measurements made on actual pipe installations (6) show an increase with time of from 1.38 to 1.46 times initial deflection. A design value of 1.25 to 1.50 is suggested for average conditions. When the internal pressure in a water pipe is equal to or exceeds the vertical pressure of the earth cover, the time-lag factor may be 1.00.

## PASSIVE DEFORMATION USING DIAGRAMS IN THIS PAPER

Figures 15 to 20 inclusive are included as aids in design and deflection control calculations. The information has been selected from published data (8,11). Other data are available. Those selected are more or less typical of remolded soils. If not otherwise specified on figures, soil specimens have been compacted to 98 Proctor density.

Figure 21 has been included for use by those designers familiar with Proctor's (9) methods of soil compaction control. The diagram represents the results of some 900 soil specimen tests. The actual range of all varieties of the remolded soils tested indicates a spread of one-half to twice the values shown. The actual consolidation of natural soils in place in the 40 to 1200 psi indicated saturation penetration resistance range may be about one-third of the values shown in Fig. 21.

If the value of $w_h$ is used in any of the Figs. 15 to 21 inclusive, or similar

$$\Delta L = \epsilon_{w_h} D \qquad \text{............} (12)$$

where the transfer pressure $w_h$ (read on vertical axis) is calculated using Eq 8. The use of the shape factor $F$ in this case is on the side of safety but seems to check well in most cases.

## RELATIONSHIP OF VERTICAL DEFLECTION TO PASSIVE DEFORMATION

Since the vertical deflection of the pipe and the shortening of the earth column are now both expressed in terms of $D$, a usable relationship exists. With $\epsilon$ expressed as a percentage change in the length of the earth column, the pipe deflection is twice that percentage. That is, if a given contact pressure causes a passive deformation corresponding to $\epsilon_{w_h} = 1$ per cent, the vertical deflection of the pipe will be 2 per cent of $D$. If the allowable vertical deflection is 5 per cent, the required passive reistance must be mobilized with a passive deformation corresponding to $\epsilon_{w_h} = 2\frac{1}{2}$ per cent.

## METHODS OF CALCULATION

*Step 1:*

The load on the pipe, which is governed by the height of cover, plus live load, impact, etc is calculated, using Eq 5. Unit earth loads in pounds per square inch for given heights of cover are given in Table II for soil weighing 125 lb per cu ft. The unit pressure for other soil weights is in direct proportion to the values given.

Intensity of live loads for H-20 and E-72 loadings are given in Table III for given height of cover.

*Step 2:*

Steel pipe for water service is usually of such diameter and wall thickness that earth loading is seldom a problem. The ring strength alone is sufficient in the case of the smaller sizes. When ring-aid in the form of active earth pressure is added to the ring strength, sizes up to about 36 in. diameter in most cases need no more than the common ordinary attention given bedding and backfilling operations. However, when the earth loads including dead weight and live load exceed the allowable ring-load plus ring-aid range, then from a design point of view the earth envelope becomes a part of the structure. Attention must therefore be given the bedding and backfilling operations to the extent that sufficient strength is built into the backfill to properly support the pipe.

TABLE II.—UNIT EARTH LOADS DUE TO HEIGHT OF COVER. SOIL WEIGHT 125 LB PER CU FT.

| Height of Cover *H*, ft | Unit Earth Load, psi | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Tens Units→ | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| 0 | ... | 0.87 | 1.74 | 2.60 | 3.47 | 4.34 | 5.20 | 6.08 | 6.94 | 7.81 |
| 10 | 8.68 | 9.55 | 10.4 | 11.3 | 12.2 | 13.0 | 13.9 | 14.8 | 15.6 | 16.5 |
| 20 | 17.4 | 18.2 | 19.1 | 20.0 | 20.8 | 21.7 | 22.6 | 23.4 | 24.3 | 25.2 |
| 30 | 26.0 | 26.9 | 27.8 | 28.6 | 29.5 | 30.4 | 31.2 | 32.1 | 33.0 | 33.9 |
| 40 | 34.7 | 35.6 | 36.5 | 37.3 | 38.2 | 39.1 | 39.9 | 40.8 | 41.7 | 42.5 |
| 50 | 43.4 | 44.3 | 45.1 | 46.0 | 46.9 | 47.7 | 48.6 | 49.5 | 50.3 | 51.2 |
| 60 | 52.1 | 52.9 | 53.8 | 54.7 | 55.6 | 56.4 | 57.3 | 58.2 | 59.0 | 59.9 |
| 70 | 60.8 | 61.6 | 62.5 | 63.4 | 64.2 | 65.1 | 66.0 | 66.8 | 67.7 | 68.6 |
| 80 | 69.4 | 70.3 | 71.2 | 72.0 | 72.9 | 73.8 | 74.6 | 75.5 | 76.4 | 77.3 |
| 90 | 78.1 | 79.0 | 79.9 | 80.7 | 81.6 | 82.5 | 83.3 | 84.2 | 85.1 | 85.9 |
| 100 | 86.8 | 87.7 | 88.5 | 89.4 | 90.3 | 91.1 | 92.0 | 92.9 | 93.7 | 94.6 |

*Ring-Load—$w_p$.*—Ring-load $w_p$ for 2 per cent deflection is given in Fig. 13 in terms of depth of cover in feet for soil weighing 125 lb per cu ft. The depth of cover may be converted to psi by referring to Table II. Figure 13 gives the height of backfill carried without ring-aid by pipes with given diameter and wall thickness. If the proposed pipe will carry the entire backfill load and any live load present, no further calculations are necessary.

Figure 13 is based upon the standard formula for an elastic ring, using a constant of 0.10 which applies to earth load on pipe in a flat-bottom trench. The constant for concentrated loads top and bottom is 0.149.

*Step 3:*

If the ring load for the pipe almost carries the vertical loads, and the internal pressure will carry the soil prism above the pipe, the engineer may elect to figure such assistance at the side of the pipe as will be given by some percentage of the active pressure of the earth in the backfill. The value of the active pressure

TABLE III.—APPROXIMATE UNIT LOADS DUE TO H-20 TRUCK AND COOPER E-72 LIVE LOADS FOR GIVEN HEIGHT OF COVER.

| Height of Cover, *H*, ft | Load, psi | |
|---|---|---|
| | H-20 | E-72[a] |
| 1 | 11 | 30 |
| 2 | 6 | 25 |
| 4 | 3 | 18 |
| 6 | 2 | 14 |
| 8 | 1 | 10 |
| 10 | 1 | 7 |
| 12 | 1 | 5 |
| 14 | ... | 4 |
| 16 | ... | 4 |
| 18 | ... | 3 |
| 20 | ... | 2 |
| 25 | ... | 1 |

[a] Loads include 50 per cent impact.

to be used must remain a matter of judgment based on circumstances of the installation. Unless the value is better known, the suggestion given previously under the definition of ring-aid may be followed. The foregoing statements presume that the trench is backfilled by ordinary methods and without tamping.

*Step 4:*

If the proposed pipe will not carry the vertical load in accordance with Eq 6, its arch-load should be determined, as given by Fig. 14. The loads given are for a compressive stress of 7,500 psi providing a safety factor of 4 for steel having a yield point of 30,000 psi and having a joint efficiency of 100 per cent in compression. Still higher loads can be carried if the designer wishes and if the installation procedures are carefully watched to provide adequate strength in the earth envelope.

*Step 5:*

Having determined the effective vertical load intensity and the ring strength of the pipe, the transfer-load from pipe to soil is determined using Eq 8.

*Step 6:*

Knowing the intensity of the transfer-load pressure, the passive deformation may be determined using Eqs 11 or 12. The value $\epsilon$ or $\epsilon_{w_h}$ is a measure of the axial strain caused by an axial load on the soil while it is at the same time being subjected to lateral radial pressures due to height of cover. It is at this point that soil mechanics must enter the problem.

## TRIAXIAL SHEAR TESTS OF SOILS

In Fig. 9 an imaginary triaxial shear test soil specimen is assumed to lie somewhere along the length of the equivalent earth column. If the position $C$ is at the contact surface of the pipe, the specimen will be subjected to the maximum axial stress existing in the equivalent earth column and the confining stress on the specimen will be that due to the depth $z$. If we then have triaxial shear test data including the stress-strain characteristics of the sample corresponding to different confining pressures due to change in depth $z$, we have the data necessary to solve Eqs 11 or 12.

Stress-strain diagrams of certain typical remolded soil materials compacted to 98 per cent maximum density are shown in Figs. 15 to 18 inclusive. They are stress-strain diagrams giving the usable axial compression in pounds per square inch corresponding to given lateral or confining pressures in pounds per square inch with axial strain given in per cent. The diagrams are presented as guides for engineering understanding and judgment rather than as solutions for particular problems. Full data on these soils and the tests conducted by the Bureau of Reclamation have been published (8) Figures 19 and 20 show similar data for graded gravel and crushed slag for maximum compaction. Data have been published (11).

*Use of Triaxial Shear Data:*

The deviator stress ordinates in Figs. 15 to 18 inclusive and the longitudinal stress ordinates in Figs. 19 and 20 are values of $w_h$ found by Eq 8 and associated with the proper applied lateral or confining pressure corresponding to distance $z$ below the surface as shown in Fig. 9. In all Figs. 15 to 18 inclusive, the abscissae (axial strain per cent) are values of $\epsilon_{w_h}$ in Eq 12 stated in per cent for associated values of $w_h$ and $z$.

## COMMENTS ON FACTORS OF DESIGN

There is a general lack of coordinated information on underground conduits of the nature necessary to analyze the problem fully. Backfilling methods are seldom described in tests and the degree of soil compaction around the pipe is rarely measured. Neither have the soil properties of the surrounding earth envelope been clearly stated. There is consequently quite a void in basic information.

### *Permissible Deflection:*

The amount of deflection to cause collapse of flexible pipe is about 20 per cent of the nominal diameter. Deflections up to 10 per cent of diameter are therefore amply safe. The spun coal-tar enamel meeting The Am. Water Works Assn. specifications for coatings and linings will safely take a 10 per cent deflection. Corrugated culvert installations are commonly designed using 5 per cent deflection. For steel water pipe, permitting 2 per cent deflection is good practice, although for large diameter pipe, unlined, deflection has sometimes amounted to as much as 10 per cent on well-engineered lines.

Permissible deflections for cement-lined pipe are on the order of 2 per cent.

Strictly speaking, $\Delta D$ is the horizontal deflection of the pipe, but within the range of use, experience has shown that it may likewise be considered to be the vertical deflection as well.

The effect on hydraulic efficiency of a pipe deflected by as much as 5 per cent is entirely negligible since its area is 99.75 per cent of that of a perfect circle and its hydraulic radius also 99.75 per cent. When the deflection is 2 per cent, the corresponding figure is 99.95 per cent for both area and hydraulic radius.

### *Vertical Loads:*

The results of many tests show that flexible steel pipe frequently carries less load than that corresponding to the weight of the prism directly over the pipe. Rigid pipe, on the other hand, has frequently shown a load greater than the weight of the prism above it. In the case of flexible pipe, some measurements have shown the full weight of the prism above the pipe transmitted to the pipe. Since carrying the full weight of the prism would probably be the worst case for flexible pipe, the full weight assumption should be used. A simple empirical means of varying the load would be to modify the estimated weight of the soil prism itself. The depth of cover, $H$, over large pipe may be taken as the average between the horizontal diameter line and the top.

### *Horizontal Pressure:*

Measurements of the horizontal pressure in flexible pipe have at times shown the horizontal intensity to be about 25 per cent more than the vertical. The shape factor used in the formula for transfer pressure intensity is intended to account for increased load due to increase in horizontal diameter, increased pressure because of shortening of vertical diameter, and increased peak horizontal intensity due to load distribution.

### *Value of $\epsilon$:*

Strain, or shortening under compression, in soil varies with such factors as void-ratio, moisture content, axial load, confining pressure, load repetition and time. The side-wise settlement or passive deformation of the soil around the pipe likewise varies with these factors.

It should be noticed that while only horizontal thrusting forces are here considered, the same general principles can apply to forces radiating from any part of the pipe periphery if the thrusting loads are known.

When important large diameter flexible pipe projects are installed, and vertical deflection is a serious factor, laboratory tests on back-fill material may be required. Field compaction control should tie field operations to laboratory data. Such field control is now common practice in the highway field on important projects and pays off in the form of better roads and lowered maintenance costs. A nominal amount of back-fill control can reduce pipe material costs on large diameter pipe installations.

One approximate method of predicting settlement is to use the Proctor indicated saturated penetration resistance in conjunction with measuring soil dry weight, both operations performed in the field. Percentage of consolidation can then be estimated using Fig. 21 (9).

## ELLIPSING, STULLING OR STRUTTING

It is common practice in the highway and railroad field to increase the vertical diameter of round pipe during installation by installing vertical columns inside the pipe. The amount of ellipsing has been a matter of judgment and experience. The method herein proposed for determining the passive deformation at the side of the pipe immediately provides an extremely simple method of determining if ellipsing is needed and to what extent the pipe diameter should be elongated vertically. Since the side-wise settlement, or increase in horizontal diameter, in the case of the pipe is similar to the deflection of a beam or bridge under load, ellipsing is the equivalent of cambering. When bridges are cambered, an upward extending bow is built into them and the amount of camber or bow is such that when loaded the beam becomes straight or the bridge floor conforms to the design contour.

The same thinking can apply to the passive deformation of the soil at the side of the pipe. For instance, if the axial strain in the soil reaches $2\frac{1}{2}$ per cent to develop a certain transfer-load· the theoretical length of the equivalent earth column can be increased $2\frac{1}{2}$ per cent by pulling the pipe side inward that amount as would be done in cambering a bridge. If then under these circumstances, the pipe is stulled or strutted 5 per cent vertically, the full transfer-load will be accepted by the two equivalent earth columns each with a $2\frac{1}{2}$ per cent passive deformation. The pipe theoretically then becomes round after installation. In this instance, if 2 per cent deflection were to be allowed in the pipe finally, 3 per cent elongation of the vertical diameter would accomplish the results. From this it can be seen that percentage of ellipsing distance is obtainable directly through the use of Figs. 15 to 21 inclusive.

The stulls or struts may be removed before backfill reaches full height if it is determined that adequate side pressure has developed. If the pipe is manufactured and installed as an ellipse with long axis vertical, no stulls or struts are necessary. Struts cannot be used with some types of field joints.

## TRENCH CONDITIONS

When pipe is put in a trench, instead of a fill, that portion of the equivalent earth column which lies between the side of the pipe and the side of the trench undergoes passive deformation which may be equal to that of the soil beyond the trench side only if the compaction between the pipe side and the side of the trench is such that it is comparable with the remainder of the length of the equivalent earth column.

When the backfill is not placed at 98 per cent of maximum density, the additional passive deformation in excess of that shown by the soil data figures may be estimated. Establish the distance $l$ between pipe wall and trench side. An extra amount of passive deformation will occur in this portion of the equivalent earth column. This additional deformation is approximately 0.5, 0.7, or 1.0 times the deformation which would be calculated for the distance $l$ for ASTM Method D 698[3] densities of 95, 93, and 90 per cent respectively. This figure is a distance and not a percentage and is to be so considered in estimating the pipe deflection.

For example, suppose a passive deformation of 1 per cent occurs at the side of a pipe whose wall is 12 in. from the trench side when the transfer pressure is developed. If this is a 48 in. pipe, the vertical deflection is 2 per cent of the diameter or 0.96 in. The compaction is to be 90 per cent standard ASTM density. The additional deflection between pipe wall and trench is $0.01(12) = 0.12$ in. for one side. The total pipe deflection is 0.96 plus twice 0.12 or 1.20 in.

It has been found that for densities below 90, ASTM Method D 698,[3] the soil resistance drops off very rapidly.

## COMPACTION CONTROL

The maximum density, as defined by ASTM Method D 698, is that obtainable with a moderate amount of rolling or tamping with the soil at optimum moisture content. The ratio of the actual soil density to the maximum density is the percentage of compaction. This indicates the degree of compaction and the ability of soil to carry heavy loads without settlement. Table IV shows classification according to compaction requirements (**12**). Data on methods of trenching and backfilling for water pipe based on a soil mechanics approach have been published (**12,13**).

## INTERNAL PRESSURE

Buried steel pipe when empty is under compressive forces due to soil weight and soil pressure. The outside vertical forces tend to flatten the pipe and create tensile and compression stresses in the pipe wall due to a decrease in vertical diameter. When internal pressure is introduced into the pipe, the first action is reversed and the internal pressure tends to force the pipe to a perfect circle. The internal pressure reduces the stresses caused by the soil. In a way of speaking, the backfilling operation has prestressed the pipe against internal pressure. For the case III design condition—full arch load and no internal pressure—the pipe wall will be under 7500 psi compressive stress if the full cover height is used and will be under proportionally lower stress for lesser heights. Whatever compressive stress exists in the pipe due to soil action first must be overcome by the tensile force due to internal pressure before the pipe wall can go into tension.

TABLE IV.—BACKFILL CLASSIFICATION ACCORDING TO SOIL COMPACTION REQUIREMENTS (**12**).

| Class | Type of Backfill | Percentage of Compaction |
|---|---|---|
| 1...... | Supporting extremely heavy loads | 100+ |
| 2...... | Subject to surface loads | 95 to 97 |
| 3...... | Not subject to surface loads | 90 to 93 |
| 4...... | Where settlement causes no harm | 85 to 90 |

For the above reason, the common practice of determining steel pipe wall thickness using the membrane theory on the basis of tensile strength to resist internal pressure is sound and conservative. It is a fallacy to total theoretical bending stresses and tensile stress for steel pipe as must be done with rigid pipe.

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[3] Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 42 T), 1955 Book of ASTM Standards, Part 3, p. 1789.

### LIVE LOAD AND SHALLOW COVER

It is obvious from the equation for intensity of contact pressure

$$W_h = 1.35\ (w_v - w_p),$$
where $F = 1.35$ in Eq 8,

that it contains a means of determining the ring strength necessary to keep the transfer pressure to its reasonable limit. Where no live loads are present, or where the depth of cover is considerable, ring strength as such is relatively unimportant. However, for live loads and shallow cover, the quantity $(w_v - w_p)$, that is, the difference between the vertical load on the pipe and its ring strength load must be such that the allowable transfer load is not exceeded. An example will illustrate the procedure of determining the wall thickness in this case.

Assume: 48 in. diameter pipe, 2 per cent deflection, 3 ft cover, H-20 live load, buried in sand-clay binder as in Fig. 16.

Then:

$$\begin{aligned} W_h &= 1.35\ (w_v - w_p) \\ w_v &= 2.6 + 5.0 = 7.6 \text{ psi} \end{aligned}$$
(Tables II and III)

From Fig. 16, the maximum $W_h$ for 1 per cent passive deformation is taken as 7.0 psi.

Then:

$$\begin{aligned} 7 &= 1.35\ (w_v - w_p), \\ 7 &= 1.35\ (7.6) - 1.35w_p, \\ 7 &- 10.26 = -1.35w_p, \text{ and} \\ w_p &= 2.4 \text{ psi.} \end{aligned}$$

From Table II, 2.4 psi corresponds to a 2.8 ft cover.

From Fig. 13, for a 2.8 ft cover, the thickness of a 48 in. pipe should be 0.375 under the live load in this soil compacted to 98 per cent density.

If there is no live load, Fig. 14 shows this pipe could be as light as 0.141 and still not deflect 2 per cent.

### EXAMPLES OF CALCULATION

*Case I—Self-Supporting Conduit:*

Case I is shown in Fig. 10(*a*) where no side support is assumed to be provided by the soil.

*Problem 1:*

What is the height of cover without live load for a 30-in. pipe having ½-in. wall thickness when allowing a maximum deflection of 2 per cent?

*Solution.*—Referring to Fig. 13, the height of cover is found to be 21 ft.

*Comment.*—Backfilling may be done with no special attention given tamping because the pipe carries the full load by ring-strength.

*Case II—Testing for Semi-Rigidity:*

Case II is shown in Fig. 10(*b*).

*Problem 2:*

Will a 30-in. pipe having a ¼-in. wall thickness sustain a 5-ft cover plus an H-20 live load without exceeding 2 per cent deflection? Backfill is sandy-clay.

*Solution: Step 1.*—Vertical load intensity equals 4.34 psi dead load (Table II) plus 2.5 psi (Table III) totaling 6.84 psi.

*Step 2.*—From Fig. 13 a dead load of 2.7 ft of cover will be carried by ring-strength. A cover of 2.7 ft is equivalent to 2.34 psi (0.1 of 23.4 psi for 27 ft cover Table II).

*Step 3.*—Is this a case II situation?

Assume ring-aid (active soil pressure) equals ⅓ of vertical soil pressure at depth of pipe axis (6.25 ft). Pressure at depth 6.25 ft (this is height $z$ Fig. 9) equals 5.4 psi (interpolated from Table II).

Assumed active pressure is $\frac{1}{3}$ of 5.4 = 1.8 psi.

$$w_r - w_a = 6.84 - 1.8 = 5.03 \quad \text{.....(6)}$$

$$\therefore \quad 5.03 > 2.34$$

Since 5.03 is greater than 2.34, this cannot be considered case II. It must be calculated as case III.

*Step 4.*—From Fig. 14 arch load is 140 ft which is more than adequate.

*Step 5.*—

$$\begin{aligned} w_h &= 1.35\,(w_{vd} + w_{el}) \\ &\qquad (F = 1.35 \text{ in Eq } 8) \\ &= 1.35\,(4.34 + 2.5) \\ &= 9.23 \text{ psi} \end{aligned}$$

*Step 6.*—

$$\Delta L = \epsilon_{w_h} D \quad \text{.............(12)}$$

From Fig. 16 it is found (by interpolation between curves 3.1 and 12.5) that for a $z$ height corresponding to 5.4 psi (see step 3) the axial strain $\epsilon_{w_h}$ for $w_h$ = 9.2 psi is about 0.8 per cent.

Then $\Delta L = 0.008(30) = 0.24$ in.

This is less than 0.01(30) = 0.30 in. allowable, so pipe will be satisfactory provided backfill is similar to that represented by Fig. 16 and is tamped to about 98 per cent of optimum density.

*Comment.*—In the foregoing example notice that there is a leeway of 25 per cent between the 0.24 in. theoretical deflection and the 0.30 in. allowable. Experience indicates this to be an ample lag factor, or time factor, to account for final soil consolidation and passive deformation.

*Problem 3—Flexible Conduit:*

A 30-in. smooth wall pipe with 0.109-in. wall thickness is to be installed with tamped sand backfill. What vertical deflection is to be expected under covers of 4, 8 and 12 ft assuming that Fig. 15 is typical of the backfill material—graded sand?

*Solution: Step 1.*—From Table II $w_{vd}$ = 3.47, 6.94 and 10.4 psi for 4, 8 and 12-ft covers respectively.

*Step 2.*—Ring-load from Fig. 13—less than 1 ft—ignore.

*Step 3.*—No ring load, no ring-aid—ignore.

*Step 4.*—Arch load from Fig. 14—by extrapolation—60 ft, and therefore satisfactory.

*Step 5.*—Transfer load, $w_h = 1.35\, w_{vd}$. $F$ = 1.35 in Eq 8.

$$\begin{aligned} w_h &= 4.68 \text{ for } H = 4 \\ &= 9.37 \text{ for } H = 8 \\ &= 14.0 \text{ for } H = 12 \end{aligned}$$

*Step 6.*—Passive deformation—Fig. 15.

| | $w_h$, psi | $z$, psi | $\epsilon w_h$, per cent | Pipe Deflection | | |
|---|---|---|---|---|---|---|
| | | | | Calculated, per cent, | Calculated, in. | Measured, in. |
| H-4 | 4.7 | 5.6 | 0.3 | 0.60 | 0.18 | 0.13 |
| H-8 | 9.4 | 10.1 | 0.6 | 0.12 | 0.36 | 0.22 |
| H-12 | 14.0 | 14.6 | 0.7 | 0.14 | 0.42 | 0.32 |

*Comment.*—Problem 3 is a duplicate, as far as is known from reports, of the Chapel Hill pipe test (**14**).

Considering the multiplicity of factors influencing the precise installation of such a flimsy pipe, plus the assumption of properties of sand, the above agreement between estimated and actual figures appears quite satisfactory for practical purposes.

*Problem 4:*

What deflection can be expected in an 84-in. diameter corrugated pipe with wall thickness of 0.281 in. installed with 137 ft cover in a 100 per cent compacted fill having properties between graded gravel in Fig. 19 and crushed slag in Fig. 20? Pipe is to be strutted 3 per cent during installation.

*Solution: Step 1.*—From Table II, $w_{vd}$ = 118.9 psi.

*Step 2.*—Ring-load. By comparison of section modulus values, this pipe has ring-load capacity about equal to 1 in. plate pipe same diameter. Then from Fig. 13, ring-load cover equals 8 ft = 6.94 psi.

*Step 3.*—Omitted.

*Step 4.*—From Fig. 14, arch load indicated over 200 ft.

*Step 5.*—Transfer load.

$$w_h\ 1.35\ (118.9 - 6.94) \qquad F = 1.35 \text{ in Eq } 8$$

$$w_h = 151.15$$

*Step 6.*—Data in Figs. 19 and 20 do not contain values for $z$ distances as great as involved here. Therefore, reading directly from diagrams: for gravel $\epsilon_{w_h} = 1.3$ per cent and for crushed slag $\epsilon_{w_h} = 0.35$ per cent.

Therefore, with gravel backfill the pipe deflection is two times 1.3 per cent or 2.6 per cent. With crushed slag the deflection is two times 0.35 per cent or 0.7 per cent. The average of the two determinations is 0.5 (3.3) = 1.65 per cent.

*Comment.*—Problem 3 postulates the conditions of the sectional bolted pipe installed and observed at Culman, Ala. (**15**). The final measured deflections on removal of struts were maximum 2 per cent and minimum about ½ per cent. The average is 1½ per cent compared with the estimated 1.65 per cent.

*Problem 5:*

A 96-in. diameter water pipe carrying 150 psi pressure to be put in a trench under 22 ft cover with no live load, allowing 2 per cent deflection.

What thickness can be used with sandy-clay backfill?

*Solution.*—Wall thickness required for internal pressure is ½ in. allowing a tensile stress of 16,000 psi.

*Solution: Step 1.*—From Table II, $w_{vd} = 19.1$.

*Step 2.*—From Fig. 13; ring-load is less than 1 ft and will be ignored.

*Step 3.*—Omitted.

*Step 4.*—From Fig. 14; arch-load is 90 ft. Since cover is only 22 ft, ½-in. wall thickness is adequate.

*Step 5.*—From Eq 8; transfer load $w_h = 1.35\ (19.1) = 25.8$ psi.

*Step 6.*—From Fig. 16; for $z = 26$ ft or 22.6 psi.

$$\epsilon w_h = 1.7 \text{ per cent}$$

$$\text{Pipe deflection} = 2\ (1.7) = 3.4 \text{ per cent.}$$

*Conclusion.*—This pipe can be held to 2 per cent deflection with fully compacted backfill of sand with clay binder provided it is strutted 3.4 − 2.0 or 1.4 per cent (about $1\frac{5}{16}$ in.).

## REFERENCES

(1) B. G. Neal, "The Plastic Methods of Structural Analysis," John Wiley and Sons, Inc., New York, N. Y. (1956).

(2) Fred B. Seely and James O. Smith, "Resistance of Materials," John Wiley and Sons, Inc., New York, N. Y. (1957).

(3) "Design and Installation of Steel Water Pipe," Manual of the American Water Works Assn., New York, N. Y. (Available 1958).

(4) "Gas Transmission and Distribution Piping Systems, Section 8 of American Standard Code for Pressure Piping" (ASA B31.1—1955), Am. Soc. Mechanical Engrs., New York, N. Y.

(5) Report on Steel Pipe Lines for Underground Water Service, Underwriter's Laboratories, Inc., Special Investigation 888 (1936).

(6) M. G. Spangler, "The Structural Design of Flexible Pipe Culverts," *Bulletin 153*, Iowa State College, Ames, Iowa (1941).

(7) Donald M. Burmister, "The Importance of Natural Controlling Conditions Upon Triaxial Compression Test Conditions," Triaxial Testing of Soils and Bituminous Mixtures, Am. Soc. Testing Mats., p. 248 (1950). (Issued as separate publication *ASTM STP No. 106.*)

(8) A. A. Wagner, "Shear Characteristics of Remolded Earth Materials," Triaxial Testing of Soils and Bituminous Mixtures, Am. Soc. Testing Mats., p. 192, (1950). (Issued as separate publication *ASTM STP No. 106.*)

(9) R. R. Proctor, "An Approximate Method for Predicting the Settlement of Foundations and Footings," *Paper No. 14.4*, Second National Conference on Soil Mechanics and Foundation Engineering, The Hague (Holland), (1948).

(10) Karl Terzaghi, "Theoretical Soil Mechanics," John Wiley and Sons, Inc., New York, N. Y., p. 380 (1943).

(11) W. S. Housel, "Interpretation of Triaxial Compression Tests on Granular Soils," Triaxial Testing of Soils and Bituminous Mixtures, Am. Soc. Testing Mats., p. 267 (1950). (Issued as separate publication *ASTM STP No. 106.*)

(12) George F. Sowers, "Trench Excavating and Backfilling," *Journal,* Am. Water Works Assn., July, 1956.

(13) Henry M. Reitz, "Soil Mechanics and Backfilling Practices," *Journal,* Am. Water Works Assn., Dec., 1956.

(14) G. M. Braune, Wm. Cain, and H. F. Janda, "Earth Pressure Experiments on Culvert Pipe," *Public Roads,* Vol. 10, No. 9, Nov., 1929.

(15) M. G. Spangler and Donald L. Phillips, "Deflections of Timber-Strutted Corrugated-Metal Pipe Culverts Under Earth Fills," *Bulletin No. 102,* Highway Research Board, *Publication 350,* Nat. Academy of Sciences, National Research Council.

## DISCUSSION

MR. E. J. WALL.[1]—We also make a flexible type of pipe, and after 25 years of research on conduits and sewer pipe, I would like to pay tribute to Mr. Barnard and his co-workers who have pioneered in the theory of loadings on flexible pipe. For information on loadings on this type of pipe, it seems that other than the Iowa State College Engineering Bulletins, you must come to Armco engineering department publications for really useful information such as the Handbook of Drainage and Construction Products.[2]

I would like some additional information. The author refers to soils in a general sense in describing earth loadings. How about extreme cases such as unstable types of soil? A couple which come to mind are the high swelling clay type such as occur in Texas and along the Gulf Coast, and also the quicksand type of soil. Would he still use the same theoretical approach outlined to calculate loadings, pipeline deflections, etc.?

Another small, but possibly important point. The theory is based on calculated loadings from the soil and strength of pipe materials within elastic limits to resist them. I would also like to visualize how a mole can burrow through the ground without the tunnel collapsing behind him. Or, how a man can dig a mine shaft tunnel that does not fall through.

By these questions I do not mean to embarrass the author. He and his organization have contributed so much to the theory that I would like to hear their views on them.

MR. RUSSELL BARNARD (*author*).—When we install conduits in squeezing clay, or quicksand, I think we take what we can but it does not pay to fight nature too far. If there are actions in squeezing clay, and loads of that kind, which we cannot handle by strength of the plate alone in an underground conduit, we would endeavor to backfill with more suitable material.

When it comes to quicksand, I have installed 6-ft diameter $\frac{1}{4}$ in. wall plate pipe under 20 ft of sand which was quick when not well pointed. The figures for deflections I have in the paper for sand check quite closely the measured deflections for that particular installation. In what would be called extreme cases, this method has checked within ½ per cent. For example, the actual deflection of a 6-ft diameter corrugated bolted sectional culvert under a 138 ft highway fill averaged 1.5 per cent of diameter. The calculated deflection averaged 1.65.

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[1] Assistant Research Director, Orangeburg Pipe Co., Orangeburg, N. Y.

[2] "Handbook of Drainage and Construction Products," Armco Drainage and Metal Products, Inc., Middletown, Ohio.

You mentioned the mole. I have often said that the bunny probably knows how to make a good strong hole, too. There are plenty of bunnies and they are not engineers but they get along all right. Of course, they may have built-in soil testing equipment. There are ways of controlling squeezing formations by allowing them to squeeze under controlled conditions but never allowing fallout to occur. If a soil moves in against a hole without fallout, it builds up around that opening a very, very dense arch, composed of minute particles. We have found in trying to resist very heavy earth loads, you have to roll with the punch. If you do not do that, you are probably in trouble.

MR. WILLIAM S. HOUSEL.[3]—I should like to have the author comment on the following question.

Are there any measurements such as referred to which checked within such close limits on soft clays at very considerable depth? In other words, it was stated that the empirical method used checks within a very small percentage under a wide variety of conditions. You seemed in your answer to the last question to make an exception in squeezing clay. Then you mentioned measurements at 130 ft depth. What kind of material was that in? What I have in mind, and I will refer to it very briefly, we have measured earth pressure in soft clay on tunnels, at a considerable depth over a period of ten years. We eventually found that if the structure involved was strong enough to carry the loads that eventually developed complete equilibrium was achieved and the pressures were maintained without change. We also had very definite indications that if a flexible structure had been used, when the time came that the full overburden pressure came on the top and bottom of the tunnel, the soil mass could not develop the required differential in vertical and lateral pressure. Consequently, if the structure had not been able to take up the balance, the presumption is, and I think the evidence is quite clear, that a flexible structure would keep on yielding and eventually complete failure would result.

Again, to restate the question, . . . do you have any experimental data that extend to soft squeezing clays at very considerable depth?

MR. BARNARD.—We have had several experiences in which you might be interested. Not too long ago, there was quite a large bolted sectional corrugated pipe installed under one of the new thruways. The bolting apparently was insufficient. The bolts sheared off in a whole line of seams allowing an overlap of from 6 in. to 1 ft. That happened not too long after the structure was in place. After the initial squeezing action occurred we have noticed nothing more.

There is another structure in which you may be interested. This one is in Canada. It was designed for installation in the type of clay which occurs around the South Saskatchewan River. A number of soils men and others collaborated in trying to design and install a structure which would just fail under the load. They selected a 60-in. diameter 20-gage corrugated pipe. . . . That is about the thickness of a dime—a thin dime. Since May 1956 it has been under a 35-ft cover in the kind of clay that moves around, squeezes, and slides. It is all right structurally even though deflected.

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[3] Professor of Civil Engineering, University of Michigan, Ann Arbor, Mich.

It has probably made use of the action I spoke of, that of allowing the soil particles to close in on the pipe, because in about three places, this 20-gage has apparently passed its yield point and has buckled the corrugations. There has been little change in shape of that structure over the past . . . year. Our thinking has been if you can get the soil to do its share, as you pointed out, and the metal to do its share, the combined action will be all right even though neither material by itself can carry the whole load.

MR. HOUSEL.—I would like to mention in connection with this discussion of the time period involved, that it took 5 years for the stresses in the soil mass to readjust themselves on the Central Avenue tunnel. Even though the tunnel structure was perfectly capable of carrying the final load after 5 yr the active earth pressure was still changing. We had readings over a period of 10 yr without too much change in the last 5 yr, so when you say turnpike construction I wonder whether the time period has been long enough to get the final answer.

# DETERMINATION OF STRAIN-HARDENING CHARACTERISTICS BY TORSION TESTING* †

By D. S. Fields, Jr.[1] and W. A. Backofen[2]

## Synopsis

Analytical considerations are presented and experimental techniques described which allow a determination of strain-hardening characteristics by torsion testing. The conventional analysis of plastic torsion in a cylindrical specimen has been generalized to include rate sensitive materials. Methods are deduced by which stress, strain, strain-rate relationships may be obtained from the fewest possible specimens with a minimum amount of graphical construction. The methods have been found to yield results identical with those obtained by other techniques.

The merits of torsion testing as a means of determining strain-hardening characteristics have been described many times (**1–4**).[3] Compared with the more frequently conducted tension or compression test, it does have two outstanding advantages: tests at constant and high deformation rate are readily made by twisting at a constant rate; and large values of strain may be reached without complications of the kind introduced when necking occurs in the tension test, or when barreling due to frictional end-effects arises in compression testing.

A major disadvantage in torsion testing has been the labor involved if the torque *versus* angle-of-twist records must be converted into shear stress-strain curves. Since the strain in a solid cylindrical specimen varies linearly from zero at the center to a maximum at the surface, values of shear stress for a strain-hardening material are obtained with the graphical analysis described by Nadai (**5**), in which the shear stress, $\tau$, at the specimen surface is given as

$$\tau = \frac{1}{2\pi a^3}\left(3M + \theta\frac{dM}{d\theta}\right) \ldots\ldots (1)$$

where:

$M$ = torque,

$dM/d\theta$ = slope of the torque-twist curve,

$a$ = specimen radius, and

$\theta$ = amount of twist in the specimen, in radians per unit length.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16–21, 1957.

† This paper is based on portions of a thesis submitted by D. S. Fields, Jr. in partial fulfillment of the requirements for the Doctor of Science degree in the Department of Metallurgy at the Massachusetts Institute of Technology.

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[2] Assistant Professor of Metallurgy, Metals Processing Laboratory, Department of Metallurgy, Massachussetts Institute of Technology, Cambridge, Massachussetts.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1271.

The corresponding surface shear strain, $\gamma$, is

$$\gamma = a\theta \quad \text{(2)}$$

The results of several detailed studies using this analysis have been reported (**2, 4, 6, 7**).

An important shortcoming of the analysis based on Eq 1 is the assumption that stress is not dependent upon strain rate. Although a valid assumption at low temperatures, appreciable error can result should the analysis be applied at high temperature when stress may be strongly dependent upon strain rate. A need for more rigorous treatment of high-temperature torque-twist data led to the relationships given below by which the temperature and rate dependence of strain hardening may be determined with a minimum of labor from torsion tests at various temperatures and twist rates. Test data for the aluminum alloy 2024-0 are presented for experimental verification of the derived relationships.

## ANALYTICAL CONSIDERATIONS

### *Determination of Torque from Shear Stress-Strain Relationships:*

Although of little practical value, this first consideration is necessary as a basis from more useful derivations to follow.

A solid cylindrical test specimen of radius $a$ may be regarded as an assembly of many thin-walled, concentric tubes. When the specimen is twisted, the torque increment, $dM$, on one such tube of radius $r$ and thickness, $dr$, is

$$dM = 2\pi r^2 \tau \, dr \quad \text{(3)}$$

where $\tau$ = shear stress at a strain of $\gamma = r\theta$ and strain rate of $\dot{\gamma} = r\dot{\theta}$, $\theta$ = twist, in radians per unit length, and $\dot{\theta} = d\theta/dt$ = twist rate.

The total torque on the specimen is

$$M = 2\pi \int_0^a \tau r^2 \, dr \quad \text{(4)}$$

Since $d\gamma = \theta \, dr$, Eq 4 may also be written:

$$M = \frac{2\pi}{\theta^3} \int_0^{\gamma_a} \tau \gamma^2 \, d\gamma \quad \text{(5)}$$

where $\gamma_a = a\theta$ is the maximum shear strain at the specimen surface. If $\tau$ is strain-rate dependent, a graphical integration of Eq 5 requires the use of several $\tau$-$\gamma$ curves, each for a different and constant value of $\dot{\gamma}$. Such curves may also be drawn to relate $\tau\gamma^2$ and $\gamma$ for constant $\dot{\gamma}$, as indicated in Fig. 1. Now the points may be defined to plot the $\tau\gamma^2$ *versus* $\gamma$ curve for the entire specimen. Corresponding values of strain and strain rate are determined at differ-

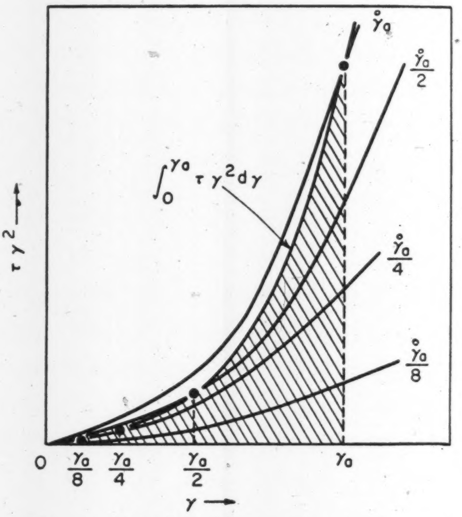

FIG. 1.—Schematic Illustration of Graphical Method Used in Computing Torque From Stress-Strain Curves.

$\tau\gamma^2$ *versus* strain for several strain rates, constructed from stress-strain curves. Shaded area represents integral quantity of Eq 5.

ent locations along the specimen radius according to the relationship:

$$\frac{\gamma}{\gamma_a} = \frac{\dot{\gamma}}{\dot{\gamma}_a} = \frac{r}{a} \quad \ldots\ldots\ldots\ldots (6)$$

Each set of values defines a point on the constant $\dot{\gamma}$ curves, and all such points define the $\tau\gamma^2$ *versus* $\gamma$ curve for the specimen, for one value of twist. As indicated in Fig. 1, the area under this curve is equal to the integral of Eq 5. A similar construction is necessary for every combination of the maximum surface strain and strain rate for which a value of specimen torque is to be determined.

### *Determination of Shear Stress from Torque-twist Relationships:*

*Tubular Specimens.*—The most direct method of determining shear stress values from the torsion test is by use of tubular specimens in which the strain and strain rate are nearly constant. If the radius to the outside surface of the tube is $a_2$ and the radius to the inside is $a_1$, the respective shear strains and shear-strain rates will be $\gamma_2 = a_2\theta$, $\gamma_1 = a_1\theta$, and $\dot{\gamma}_2 = a_2\dot{\theta}$, $\dot{\gamma}_1 = a_1\dot{\theta}$, after twisting by an amount $\theta$ at rate $\dot{\theta}$. With $\gamma_1$ as the lower limit in Eq 5, the torque on the specimen is

$$M = \frac{2\pi}{\theta^3} \int_{\gamma_1}^{\gamma_2} \tau\gamma^2 \, d\gamma \quad \ldots\ldots\ldots (7)$$

The average value of $\tau$ between the limiting strains of $\gamma_1$ and $\gamma_2$ is $\bar{\tau}$. If $a_2$ is not much larger than $a_1$, the differences between $\gamma_1$ and $\gamma_2$, and $\dot{\gamma}_1$ and $\dot{\gamma}_2$, are small so that $\bar{\tau}$ may be considered constant and Eq 7 integrated directly:

$$M = \frac{2\pi\bar{\tau}}{\theta^3}\left(\frac{\gamma_2^3 - \gamma_1^3}{3}\right) \quad \ldots\ldots (8)$$

Substituting for $\gamma_2$ and $\gamma_1$, the torque equation for the tubular specimen may be written to show that

$$\bar{\tau} = \frac{3M}{2\pi(a_2^3 - a_1^3)} \quad \ldots\ldots (9(a))$$

The corresponding average shear strain and strain rate are

$$\bar{\gamma} = \frac{a_1 + a_2}{2}\theta; \qquad \bar{\dot{\gamma}} = \frac{a_1 + a_2}{2}\dot{\theta} \ldots (10)$$

*Differential Testing.*—The use of tubular specimens may pose experimental problems. It is possible, however, to determine a torque value equivalent to that on a tubular specimen by twisting two solid specimens of radii $a_2$ and $a_1$. Upon twisting an amount $\theta$, the difference in torques on the two specimens $(M_2 - M_1)$ is equal to the torque, $M$, in Eq 9($a$), so that the shear stress may be expressed as

$$\bar{\tau} = \frac{3}{2\pi}\left(\frac{M_2 - M_1}{a_2^3 - a_1^3}\right) \quad \ldots\ldots (9(b))$$

Such a method of determining shear stress has been termed differential torsion testing, and has been used previously for a limited number of room-temperature tests (**8**).

*Solid Specimens.*—As noted above, stress may be computed from the torque-twist records of single specimens according to Eq 1 only when the stress level is insensitive to strain rate. The established analysis based on Eq 1 may be applied, however, even when rate sensitivity is a consideration, if $(dM/d\theta)$ is determined from an $M$-$\theta$ plot constructed from the results of tests at many twist rates. The problem now is similar to that involved in evaluating the integral of Eq 5. The procedures to be followed are developed in the following way:

A solid specimen of radius $a$ is considered to consist of two parts, a solid core of radius $r$ within a tube of outer radius $a$ and inner radius $r$. According to Eq 5, the torque $M_a$ on this specimen after twisting an amount $\theta_a$ at a rate $\dot{\theta}$, is

$$M_a = \frac{2\pi}{\theta_a^3}\int_0^{\gamma_a} \tau\gamma^2\,d\gamma$$

$$= \frac{2\pi}{\theta_a^3}\left(\int_0^{\gamma_r} \tau\gamma^2\,d\gamma + \int_{\gamma_r}^{\gamma_a} \tau\gamma^2\,d\gamma\right) \ldots\ldots (11)$$

where:
$\gamma_a = a\theta_a$,
$\gamma_r = r\theta_a$, and $\dot{\gamma}_a = a\dot{\theta}_a$ and $\dot{\gamma}_r = r\dot{\theta}_a$ are the corresponding strain rates.

Now, a second specimen, also of radius $a$, is twisted, but by a smaller amount $\theta_b$ and at a lower rate $\dot{\theta}_b$. The values of $\theta_b$ and $\dot{\theta}_b$ are selected so that at the surface of this specimen the strain is $\gamma_b = a\theta_b = r\theta_a = \gamma_r$—the strain at the surface of the core in the first specimen—and the strain rate is $\dot{\gamma}_b = a\dot{\theta}_b = r\dot{\theta}_a = \dot{\gamma}_r$. Therefore, the torque, $M_b$ on the second specimen is

$$M_b = \frac{2\pi}{\theta_b^3}\int_0^{\gamma_r} \tau\gamma^2\,d\gamma \ldots\ldots (12(a))$$

and

$$\int_0^{\gamma_r} \tau\gamma^2\,d\gamma = \frac{\theta_b^3 M_b}{2\pi} \ldots\ldots (12(b))$$

The integral quantity in Eq 12($b$) is identical to that appearing in Eq 11; both integrations are performed over the same range of strain and strain rate. Therefore, Eq 11 may be rewritten:

$$M_a = \frac{\theta_b^3}{\theta_a^3} M_b + \frac{2\pi}{\theta_a^3}\int_{\gamma_b}^{\gamma_a} \tau\gamma^2\,d\gamma \ldots\ldots (13)$$

The difference in torque between the two specimens $(M_a - M_b)$, is found by subtracting $M_b$ from both sides of Eq 13:

$$M_a - M_b = \left(\frac{\theta_b^3 - \theta_a^3}{\theta_a^3}\right) M_b + \frac{2\pi}{\theta_a^3}\int_{\gamma_b}^{\gamma_a} \tau\gamma^2\,d\gamma \ldots (14)$$

When $r$ (of the first specimen) approaches $a$ as the limit, $M_b \rightarrow M_c$, $(M_a - M_b) \rightarrow dM$, $(\theta_b^3 - \theta_a^3)/\theta_a^3 \rightarrow -d(\theta^3)/\theta^3 = -3d\theta/\theta$, and the integral in Eq 14 approaches the integrand $\tau\gamma^2\ d\gamma = \tau a^3\theta^2\ d\theta$. Introducing these changes into Eq 14 leads to

$$dM = -\frac{3M\,d\theta}{\theta} + \frac{2\pi\tau a^3\,d\theta}{\theta} \ldots\ldots (15)$$

or

$$\tau = \frac{1}{2\pi a^3}\left(3M + \theta\frac{dM}{d\theta}\right) \ldots\ldots (16)$$

Equation 16 is identical in form to Eq 1, but it is not restricted to rate insensitive materials if $dM/d\theta$ is obtained from a torque-twist curve which satisfies the condition under which the derivation was made. The condition, restated, was that $a\theta_b = r\theta_a$ and $a\dot{\theta}_b = r\dot{\theta}_a$, or

$$\frac{\theta_b}{\dot{\theta}_b} = \frac{\theta_a}{\dot{\theta}_a} \ldots\ldots\ldots\ldots (17(a))$$

Such a condition requires that, if shear stress values are to be obtained from a single torque-twist record, the twist rate must vary in proportion to the amount of twist:

$$\dot{\theta} = \theta\left(\frac{\dot{\theta}_a}{\theta_a}\right) \ldots\ldots\ldots\ldots (17(b))$$

where $\dot{\theta}_a$ is the twist rate at some reference value of twist, $\theta_a$. Since $\gamma_a = a\theta_a$ and $\dot{\gamma}_a = a\dot{\theta}_a$, the values of shear stress determined by application of Eq 16 to this type of torque-twist record would be associated with a different $\dot{\gamma}$ for each value of $\gamma$. Any use of such records would require the assumption that $\tau$ depends only upon $\gamma$ and the instantaneous value of $\dot{\gamma}$. In general, however, this assumption is not valid, for $\tau$ is also influenced by strain-rate history (9).

The desired $M$-$\theta$ record must, therefore, be constructed with tests at constant $\dot{\theta}$. Then a composite curve relating the "constructed" torque, $M_c$, and

$\theta$ or $\gamma$ may be determined by selecting the torque level for any value of $\theta$ at the twist rate, $\dot{\theta}$, required to satisfy Eq 17. Such a curve is illustrated in Fig. 2. With the constructed curve, values of $\tau$ may be calculated from Eq 16. The labor involved, however, could be considerable, since the resulting $\tau$-$\gamma$ curve relates $\tau$ and $\gamma$ for $\dot{\gamma}$ increasing in proportion to $\gamma$. A family of constructed $M_c$-$\theta$ curves would be needed, each representing a different, constant value of $\dot{\theta}/\theta$; the corresponding family of $\tau$-$\gamma$ curves that would be possible from such data could then be cross-plotted to yield $\tau$-$\gamma$ curves for constant $\dot{\gamma}$.

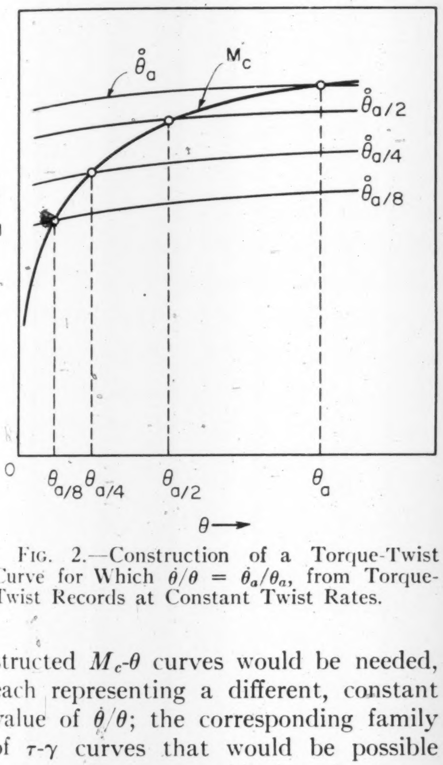

FIG. 2.—Construction of a Torque-Twist Curve for Which $\dot{\theta}/\theta = \dot{\theta}_a/\theta_a$, from Torque-Twist Records at Constant Twist Rates.

At first sight, the technique of differential testing would appear much less time-consuming than the procedures outlined above. However, it is possible to obtain a sufficient number of values of $\tau$ from few tests by taking advantage of the fact that plots on logarithmic coordinates of $M$ *versus* $\theta$ at constant $\dot{\theta}$, and of $M$ *versus* $\dot{\theta}$ at constant $\theta$ are often straight, or nearly straight, lines.

Considering a constant-temperature $M$-$\theta$-$\dot{\theta}$ surface obtained from tests at various constant rates of twist, the change in torque due to a small change in $\theta$ may be expressed as

$$\frac{dM}{d\theta} = \left(\frac{\partial M}{\partial \theta}\right)_{\dot{\theta}} + \left(\frac{\partial M}{\partial \dot{\theta}}\right)_{\theta} \frac{d\dot{\theta}}{d\theta} \quad \ldots\ldots (18)$$

If $d\dot{\theta}/d\theta$, which defines the change in twist rate, $d\dot{\theta}$, during an increment of twist, $d\theta$, is consistent with Eq 17($b$), $dM/d\theta$ of Eq 18 becomes $dM_c/d\theta$. According to Eq 17($b$), $\dot{\theta} = \theta(\dot{\theta}_a/\theta_a)$; therefore, it is necessary that

$$\frac{d\dot{\theta}}{d\theta} = \frac{\dot{\theta}_a}{\theta_a} = \frac{\dot{\theta}}{\theta} \quad \ldots\ldots\ldots\ldots (19)$$

Substituting into Eq 18:

$$\frac{dM_c}{d\theta} = \left(\frac{\partial M}{\partial \theta}\right)_{\dot{\theta}} + \left(\frac{\partial M}{\partial \dot{\theta}}\right)_{\theta} \left(\frac{\dot{\theta}}{\theta}\right) \quad \ldots (20)$$

and since $d(\ln\ x) = dx/x$:

$$\frac{dM_c}{d\theta} = \frac{M}{\theta}\left(\frac{\partial \ln M}{\partial \ln \theta}\right)_{\dot{\theta}} + \frac{M}{\dot{\theta}}\left(\frac{\partial \ln M}{\partial \ln \dot{\theta}}\right)_{\theta}\left(\frac{\dot{\theta}}{\theta}\right)$$

or

$$\frac{dM_c}{d\theta} = \frac{M}{\theta}(n + m) \quad \ldots\ldots\ldots (22)$$

where $n$ and $m$ are, respectively, the slopes of the plots of $M$ *versus* $\theta$ at constant $\dot{\theta}$, and of $M$ *versus* $\dot{\theta}$ at constant $\theta$, on logarithmic coordinates.

A final substitution of Eq 22 into Eq 16 results in an expression for $\tau$ which incorporates the condition stated in Eq 17:

$$\tau = \frac{M}{2\pi a^3}(3 + n + m) \quad \ldots\ldots (23)$$

## MATERIAL AND PROCEDURES

The aluminum alloy 2024-0 was used in the experimental examination of certain of these analyses. All test material was fabricated from a single ingot to minimize property variations due to differences in chemical composition and processing history.

The initial objective was to assess the usefulness of the differential testing technique. For each temperature-rate combination, three specimens of slightly different diameter were tested. At temperatures of 500, 350, and 200 C, specimen diameters were 0.550, 0.500, and 0.450 in. At 28 and −195 C the diameters were 0.450, 0.420, and 0.390 in. Strain rates employed were $\dot{\gamma}$ = 24, 6, 0.96, 0.16, and 0.06 per min.

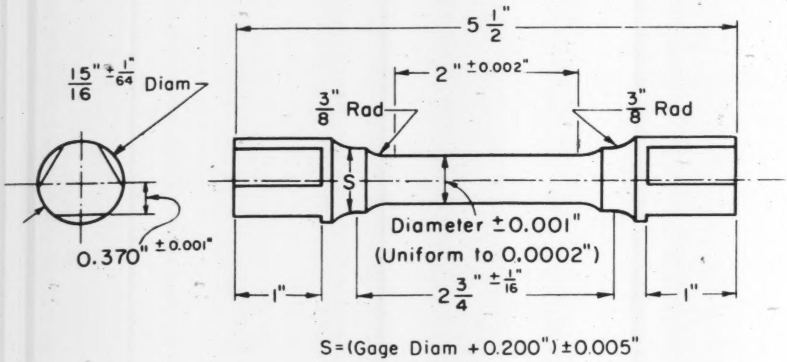

FIG. 3.—Specimen Design.

From the torque-twist records for the largest and smallest specimens, shear stresses and strains were calculated with Eqs 9(*b*) and 10. Records from the third specimen of intermediate size were useful in estimating reproducibility of data as described in the Appendix. It was concluded that torque was reproducible to ±1 per cent, while the stress, from the differential procedure reported here, was reproducible to ±3 per cent.

Records from the intermediate specimen were also used in shear stress calculations based on Eq 23, which are compared with the results of the differential analysis.

Testing was carried out in a specially constructed machine instrumented to provide autographic records of torque *versus* total angle of twist, capable of operating at a maximum speed of 30 rpm, and at torques up to 1500 in-lb. Various twist rates were obtained by insertion of combinations of gear reducers in the drive train of the upper grip. Specimens were held in a vertical position, and for tests at 200 to 500 C were immersed in a controlled-temperature bath containing approximately equal parts of sodium nitrite and of sodium and potassium nitrate, with an addition of potassium dichromate. Specimens were immersed in liquid nitrogen for tests at −195 C. Temperature differences in the gage section were measured to be less than 0.25 C.

Design of the specimens is shown in Fig. 3. Very close tolerances were maintained in machining the gage section to insure that twisting would be uniform, and that specimens to be compared according to Eqs 9 and 10 would be of identical gage length. To minimize residual cold-work in the gage section, successively lighter cuts were taken, and the surface was finally polished with 0, 00, and 000 emery paper wet with kerosene.

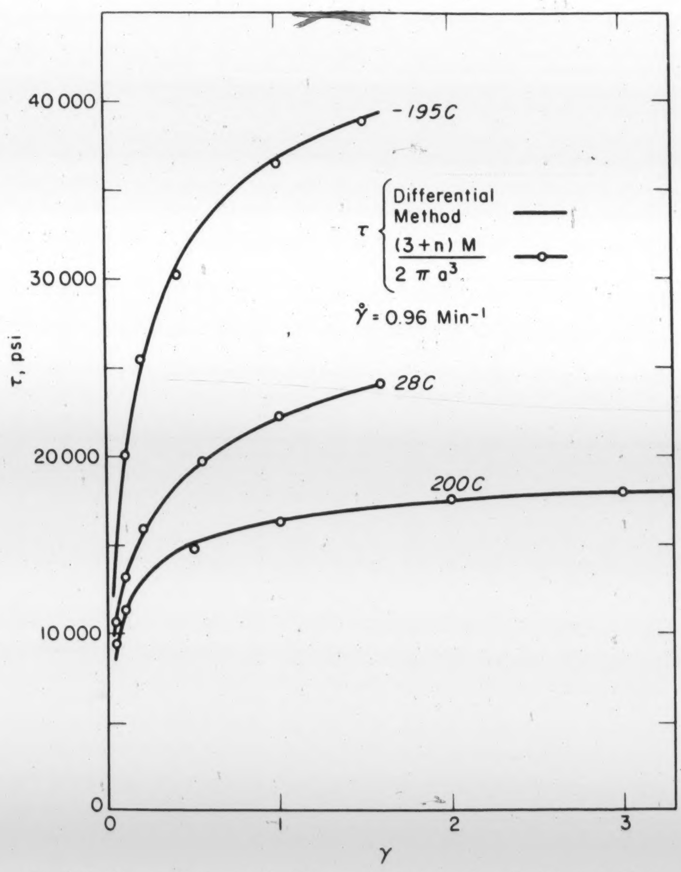

FIG. 4.—Shear Stress-strain Curves for Comparing the Two Methods of Treating Torsion Test Data.

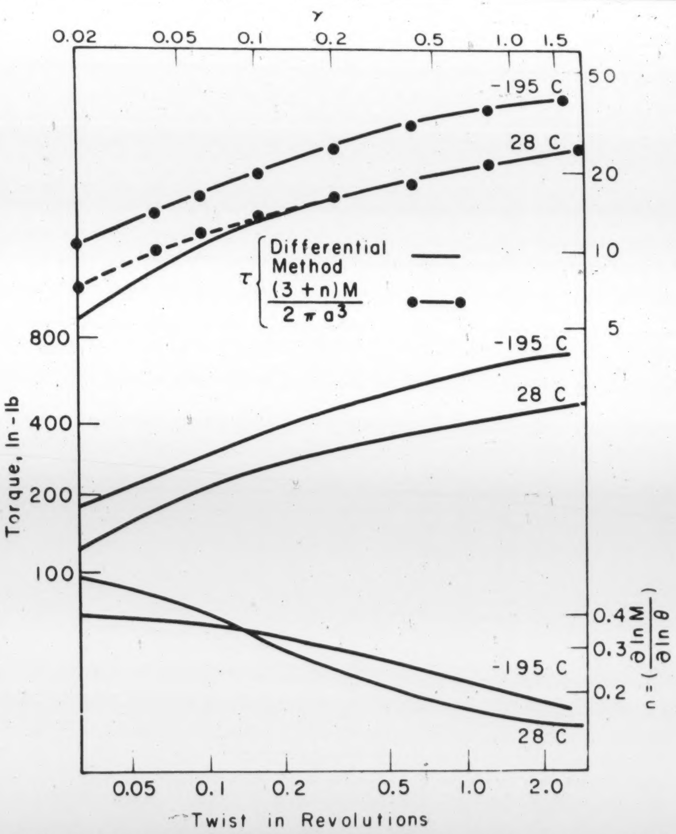

FIG. 5.—Logarithmic Plots Showing Development of Stress-Strain Curves From Torque-Twist Records When Rate Sensitivity is Insignificant; $\dot{\gamma} = 0.96$ per min.

Upper: Shear stress-strain curves determined by differential torsion (solid lines) and from curves below (plotted points).

Middle: Experimental torque-twist curves.

Lower: Slopes of torque-twist curves *versus* amount of twist.

The torque-meter design was based on a four-arm, wire resistance strain-gage bridge. Dead-load calibrations were made periodically, and internal calibration was made before and after each test. At all levels, torque could be measured to within ±0.5 per cent. Angle of twist was determined from the rotation of the upper grip with an accuracy of 0.003 revolutions.

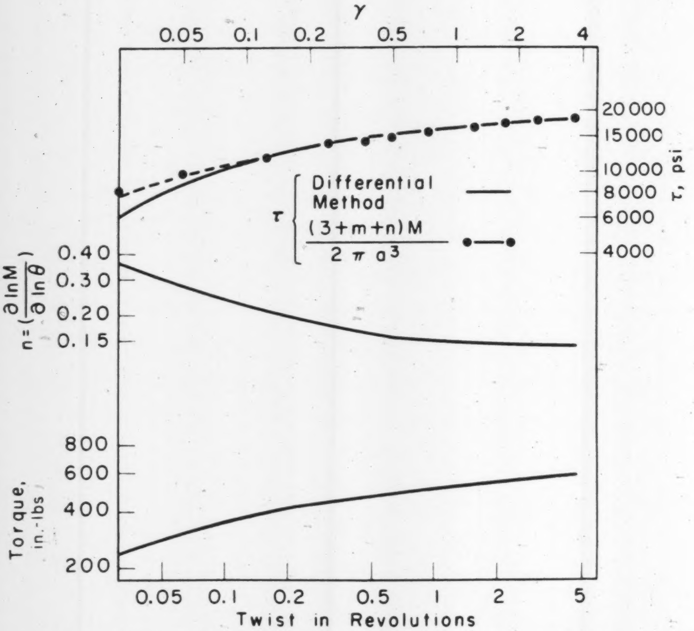

FIG. 6.—Logarithmic Plots Showing Development of Stress-Strain Curve From Torque-Twist Records at 200 C, When Rate Sensitivity is Significant; $\dot{\gamma}$ = 0.96 per min.

Upper: Stress-strain curve determined by differential torsion method (solid lines) and from curves below (plotted points). Variation of $m$ with strain determined from curves such as those in Fig. 7.

Middle: Change in slope of torque-twist curve with amount of twist.

Lower: Experimental torque-twist record.

## RESULTS AND DISCUSSION

### *Interpretation of Data:*

Stress-strain curves in shear, obtained by differential testing at several temperatures, are presented in Figs. 4, 5, and 6. The continuous curves in Fig. 4 are plotted with coordinates linear in $\tau$ and $\gamma$, while those in Figs. 5 and 6 are plotted on logarithmic coordinates. The upper curves of Fig. 7 show the variation in shear stress at $\gamma = 1.0$ with strain rate. At 350 and 500 C, shear stress depended only slightly upon strain, so that the curves in Fig. 7 depict the complete $\tau$-$\gamma$-$\dot{\gamma}$ behavior at these temperatures.

The differential test results presented were derived with records from approximately 50 specimens. Aside from the care necessary in machining specimens to the required tolerances, the most labor was involved in interpreting test records accurately; as discussed in the Appendix, scatter in torque measurements is magnified in determination of shear stress by the differential method.

The plotted points on the $\tau$-$\gamma$ and $\tau$-$\dot{\gamma}$ curves of Figs. 4 through 7 represent values calculated with Eq 23. Requiring only one test for each testing condition, these points were obtained with the records from only 16 specimens. The results by the second method were in excellent agreement with those from differential torsion. Furthermore, since stress-strain values were determined with the records of single tests, reproducibility is improved, being approximately equal to that of the torque values, or ±1 per cent.

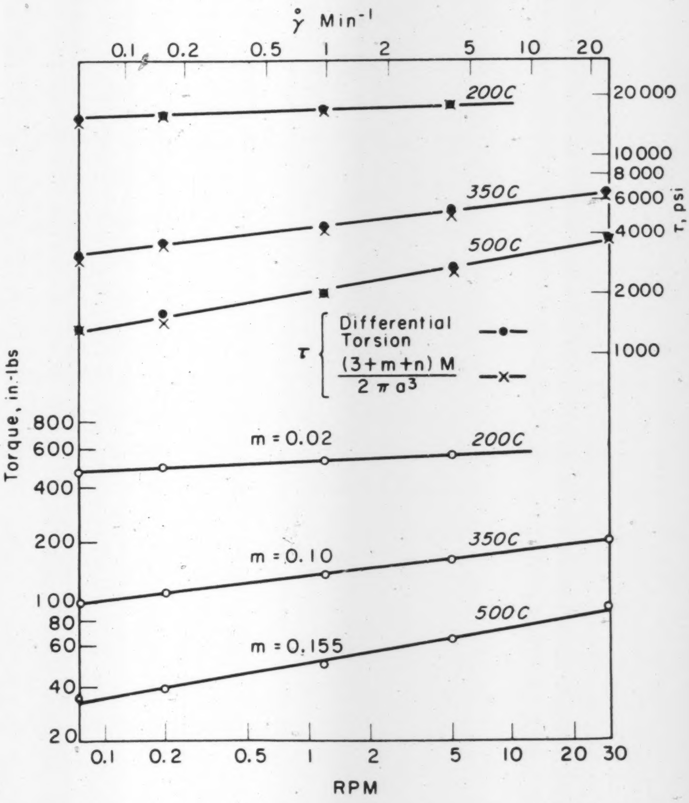

FIG. 7.—Logarithmic Plots Showing Development of Stress - Strain-Rate Curves at a Strain of 1.0 From Torque-Twist Records.

Upper: Stress - strain-rate plots determinee by differential method and from curves below. $n$ at 350 and 500 C = 0;

Lower: Torque, twist-rate plots.

The shear stress, strain, strain-rate relationships obtained with Eq 23 were determined from torque, twist, twist-rate data falling into one of three categories with respect to the relative values of $n$ and $m$, and, as a consequence, with respect to the steps required in calculating values of shear stress. The categories, and sample calculations leading to the results presented, are described below.

1. *Low temperatures: m is negligible.*—At −195 and 28 C, values of $m$ were of the order of a few thousandths, and therefore could be overlooked in calculations of shear stress. Now the conventional analysis leading to Eq 1 could be applied, but since values of $n$ are

more nearly uniform than those of $(dM/d\theta)$, the technique described is considered to be less tedious and more accurate. As shown in Fig. 5, the raw torque twist data were plotted on logarithmic coordinates, and the curves of $n$ *versus* twist were obtained from the slopes.[4] Then, from corresponding values of $M$ and $n$, shear stresses were calculated according to Eq 23 with $m$ being zero.

The slight divergence at low strains at 28 C, shown in Fig. 5, between the points from the above calculation and from differential results is due to the fact that the differential results represent average values of $\tau$ over a range of strains, and since the slope of the $\tau$-$\gamma$ curve at low strains is changing rapidly, the average value might be somewhat less than the true value. The divergence is not serious, however, since the range of strain over which it occurs is quite small.

2. *High temperatures: n is negligible.*—At 350 and 500 C, torque varied only slightly with the amount of twist, so that $n$ was negligible. As seen in Fig. 7, however, $m$ was sizable, although its value at either temperature was independent of the twist rate. It was found further that $m$ was virtually independent of the amount of twist. Therefore, at 350 and 500 C the values of $\tau$ plotted in Fig. 7 were obtained by substitution of the values of $M$ and $m$ into Eq 23 with $n$ set equal to zero.

3. *Intermediate temperatures: n and m are both significant.*—Interpretation of test results at 200 C required the most effort, since both $m$ and $n$ were significant and varied with strain. To construct the $\tau$-$\gamma$ curves at 200 C in Figs. 4 and 6, the curve relating $n$ to amount of twist was first established from the torque-twist curve shown in Fig. 6. Then, values of $m$ were determined from torque, twist-rate plots such as the one shown in Fig. 7; since $m$ varied with strain, a value was necessary for each calculated point on the stress-strain curve. Having established $n$ and $m$ as a function of strain, the $\tau$-$\gamma$ curve for $\dot{\gamma} = 0.96$ per min, plotted in Figs. 4 and 6, could be constructed. The agreement with that from differential testing was again very good.

To construct $\tau$-$\gamma$ relationships at other strain rates, a curve of $n$ *versus* amount of twist would be needed for each additional strain rate. Such curves, used in conjunction with the established curve relating $m$ and amount of twist, permit the construction of other $\tau$-$\gamma$ curves. In the example under discussion, $n$ was essentially the same for all values of $\dot{\gamma}$ at $\gamma = 1.0$, so that the $\tau$-$\dot{\gamma}$ curve of Fig. 7 could be computed from single values of $n$ and $m$.

The procedures outlined above should be adequate for obtaining relationships for almost all metals and alloys. Calculations will be rarely more complicated, and often more simple, than those described. Logarithmic plots of torque *versus* twist rate almost invariably result in straight lines; then, as for the aluminum alloy, a single value of $m$ will be required in the determination of stress at all strain rates, for a given temperature and value of $\gamma$. Further, cases will be found, as at 350 C, in which $m$ is not strain-dependent, and only one value will be required at a given temperature. Finally, there will be instances when logarithmic torque-twist curves at low temperatures are linear, and a single value of $n$ will be sufficient for calculating a complete stress-strain curve.

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[4] Slopes of curves plotted on logarithmic coordinates are not changed if all ordinate or abscissa values are multiplied by a constant. Since $\ln x = 2.3 \log x$, and since multiplication of total twist in revolutions by $2\pi$ (gage length of specimen) results in $\theta$ in radians per unit length, it is possible to obtain $\ln M$ *versus* $\ln \theta$ and $\ln M$ *versus* $\ln \dot{\theta}$ slopes, respectively, from plots relating $M$ to twist in revolutions and to rpm on common logarithmic coordinates. As no conversions are required in transcribing test results to the graph paper by this system, it is the one recommended and employed in Figs. 5, 6, and 7.

*Instability:*

For torsion test data to be meaningful, it is essential that the specimen undergo no localized deformation, or exhibit no instability. Certain conditions, however, promote instability, and then values of strain and strain rate calculated from the amount and rate of twisting will be much smaller than those in the localized region being deformed.

To illustrate factors influencing instability, a number of 0.500-in. diameter specimens were prepared with a slight taper to a 0.495-in. diameter at the middle of the gage section. Tendencies toward instability would be exaggerated by this design. After twisting, the actual strain along the surface of the gage section was determined from the angle $\alpha$ included between the specimen axis and helical deformation markings in the surface. Such markings rotated during twisting in the manner of reference

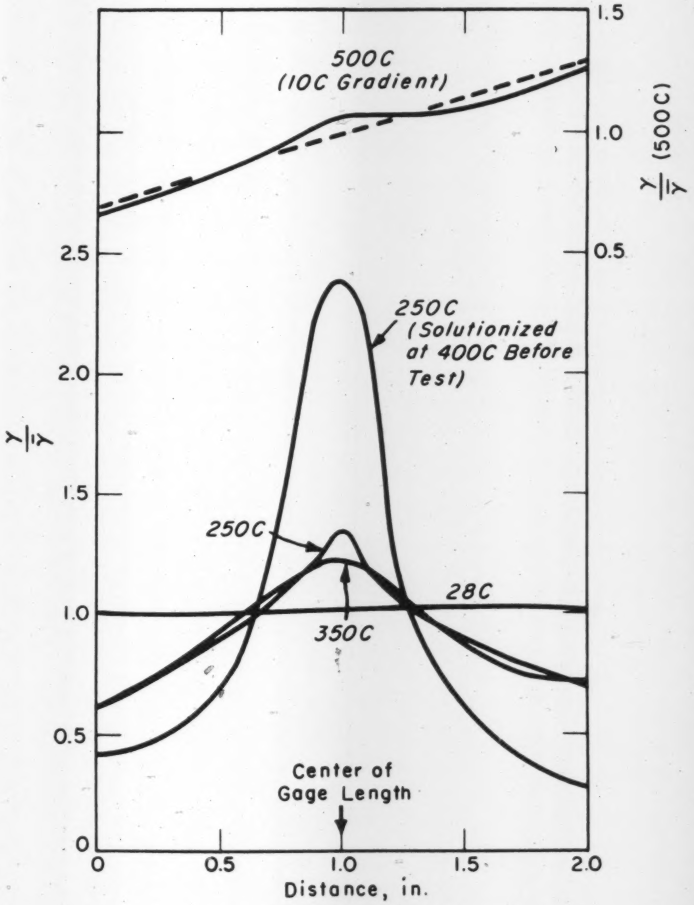

FIG. 8.—Effect of Temperature on Uniformity of Twisting in Slightly Tapered Specimens. Ratios of measured strain, $\gamma$, to average strain $\bar{\gamma}$ along gage section tapering from 0.500-in. diameter to 0.495 in. at center.

marks initially inscribed parallel to the specimen axis. In Fig. 8, the ratio of measured strain, $\gamma = \tan \alpha$, to the average strain, $\bar{\gamma}$, computed from the number of revolutions of twist, is plotted along the gage section for several conditions of twisting.

The two factors which contribute to uniform deformation and the absence of instability are high rates of strain hardening and high strain-rate sensitivity. Each is illustrated in Fig. 8 by test results at 28 and 500 C, respectively. The strain-hardening rate at 28 C was sufficiently high over the range of strain investigated that the very small increment of additional deformation in the reduced section was adequate to bring the strength of the test specimen to a uniform level along the gage length. In testing at 500 C, the salt bath was not stirred in order to establish a temperature gradient. The temperature difference between the ends of the gage section was 10 C, and its presence is reflected in a variation in $\frac{\gamma}{\bar{\gamma}}$. A gradient in temperature would be a factor promoting instability, yet a true instability has not resulted in the present example. The variation in $\frac{\gamma}{\bar{\gamma}}$ is perfectly consistent with the change in strength level accompanying the uniform gradient in temperature; both the strain and strain rate at the center of the gage length were equal to the average values. The absence of instability even when there is no strain hardening arises from the high rate-dependence of flow stress at this general temperature level. Any localized deformation would have resulted in higher strain rates. The higher rates would have meant higher values of flow stress, so that localized deformation would have ceased. In this way, the onset of instability was resisted.

The combination of low strain-hardening rate and strain-rate sensitivity would, of course, promote instability. Illustrations are provided by test results at 250 and 350 C. After heating at 400 C followed by quenching, the rate of strain hardening is extremely low at 250 C, and rate sensitivity is also low. It was impossible to produce uniform twisting under such conditions, even with highly uniform specimens and the closest control of temperature. The alloy in the annealed state is characterized by generally higher strain-hardening rates at 250 C, so that the variation in $\frac{\gamma}{\bar{\gamma}}$ is less pronounced. At 350 C, rate of strain hardening is negligible, but now rate sensitivity is higher. The result is much like that for the annealed alloy at 250 C.

## SUMMARY

Two methods have been described by which stress-strain relationships may be obtained from torsion tests when flow stress is rate sensitive. The first is termed differential torsion, which provides the average stress in a tubular specimen by calculation from the torque difference between two solid specimens of slightly different diameters. The second results from a generalization of the conventional analysis for a solid, cylindrical specimen. The second method is the more useful of the two, as it permits the calculation of the flow stress as a function of strain and strain rate with a minimum number of specimens by using the slopes of logarithmic torque, twist, twist-rate plots and selected values of torque.

Strain-hardening characteristics of the aluminum alloy 2024-0 have been determined by each of the two methods for broad ranges of temperature and strain rate. Results by the two methods are virtually identical, with the second providing somewhat greater accuracy.

A series of tests is presented which illustrates conditions controlling the onset of instability or localized deformation in the twisting of cylindrical specimens.

*Acknowledgment:*

The authors are indebted to the Aluminum Company of America for its support of this work.

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## REFERENCES

(1) C. E. Work and T. J. Dolan, "The Influenee of Temperature and Rate on the Properties of Metals in Torsion," Engineering Experiment Station, Bulletin Series No. 420, University of Illinois, November, 1953.

(2) Ya. B. Fridman, "Mechanical Testing of Materials by the Torsion Method," *Metallurgia*, Vol. 37, Nov. 1947, p. 53; abstracted from *Zavodskaya Laboratoriya*, Vol. 11, No. 9, p. 852 (1945).

(3) F. G. Tatnall, "Use of the Torsion Test," *Instruments*, Vol. 23, June 1950, p. 564.

(4) M. C. Fetzer, "Torsional *versus* Tensile Properties of Steels," *Steel*, Vol. 118, No. 25, June 1946, p. 92.

(5) A. Nadai, *Theory of Flow and Fracture of Solids*, McGraw-Hill Book Co., Inc., New York, N. Y., p. 349 (1950).

(6) H. Larsen and E. R. Klier, "Strain Hardening of Mild Steel in Torsion Tests as a Function of Temperature," *Transactions*, Am. Soc. Metals, Vol. 43, p. 1033 (1951).

(7) R. D. Olleman, E. T. Wessel, and F. C. Hull, "A Study of Factors Controlling Strength in the Torsion Test," *Transactions*, Am. Soc. Metals, Vol. 46, p. 87 (1954).

(8) N. H. Cook, Private Communication.

(9) J. E. Dorn, A. Goldberg, and T. E. Tietz "The Effect of Thermal-Mechanical History on the Strain Hardening of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 180, p. 205 (1949).

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## APPENDIX

## ESTIMATE OF REPRODUCIBILITY

An indirect method was used to estimate the reproducibility of shear stress values from differential testing. For most of the test program, shear stresses were determined from the torque differences between specimens of 0.550 and 0.450-in. diameter. The torque on the larger specimen was always about twice that on the smaller. Therefore, if $\pm x$ is the fractional reproducibility of a torque measurement, and the torque on the smaller specimen is $M(1 \pm x)$, the torque at the same twist on the larger specimen will be $2M(1 \pm x)$. Since variations of both material and experimental origin may be additive, the torque difference is

$$\Delta M_1 = M(1 \pm 3x)$$

which, according to Eq 9(*b*), is proportional to the shear stress.

To estimate $x$, a third specimen of 0.500-in. diameter was twisted. Torque for the given amount of twist was approximately $(3/2)M(1 \pm x)$, so that the torque differences between this specimen and the other two are

$$\Delta M_2 = (M/2)(1 \pm 7x)$$

and

$$\Delta M_3 = (M/2)(1 \pm 5x)$$

Next, shear stress values were determined with Eq 9(*b*) from $\Delta M_2$ and $\Delta M_3$. Then, the fractional differences in these values at various strains for each temperature-rate combination were computed and averaged. The quantity so determined lay in the range of $5x$ to $7x$; by assuming an extreme of $5x$, the maximum value of $x$ was determined to be 1 per cent. Since all differential shear stress-strain curves presented in this paper were computed using $\Delta M_1$, reproducibility is reported to be $\pm 3$ per cent.

## DISCUSSION

MR. M. J. MANJOINE.[1]—As the test temperature was increased, were any torque-wist curves encountered in which the maximum torque occurred below a strain of 1?

MR. D. S. FIELDS, JR. (*author*).—Only the aluminum alloy, 2024, has been studied thoroughly. In completely annealed specimens, torque maxima were not observed in tests at temperatures below 200 C. At higher temperatures, the maximum torque usually was reached at a surface shear strain of 1.2 to 1.5 and was followed by an extremely gradual decrease in torque with additional twisting. At an intermediate speed at 350 F, for example, the maximum torque was reached at $\gamma = 1.25$; at $\gamma = 4.0$, the torque had dropped less than 2 per cent, and at $\gamma = 10$, less than 3 per cent from the maximum value. Only in specimens that were metallurgically unstable at the testing temperature were pronounced maxima of torque obtained. In most such cases, the torque reached its maximum value at an extremely low strain, and dropped sharply as testing progressed. As discussed in the paper, this apparent strain softening was invariably accompanied by localized twisting in the torsion specimen.

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[1] Westinghouse Electric Corp., Pittsburgh, Pa.

# A LAMINATED SPECIMEN FOR CHARPY IMPACT TESTING OF SHEET METAL

By S. V. Arnold[1]

## Synopsis

A specimen has been developed which permits measure of sheet metal toughness over a range of temperatures using conventional pendulum-type testing apparatus and test procedures. Blanks are cut from the sheet and riveted together to form a laminate from which the specimen is machined to dimensions following those of the standard Charpy V-notch design. Toughness of the sheet is calculated by dividing the impact strength of the laminated specimen by the number of laminae which it contains. Tests have shown that the specimen yields highly reproducible data and is sensitive to metallurgical variables affecting toughness.

Test of specimens fashioned from laminae machined from steel plate disclosed linear relations for (*a*) the impact transition temperature and (*b*) the minimum energy of an individual lamina in ductile fracture *versus* thickness of an individual lamina. This linearity maintained over a range of thickness from 0.050 in. to 0.200 in.

Test of laminated specimens prepared from titanium alloy sheet of various compositions indicated that impact strength for a given composition at a selected temperature is closely proportional to sheet thickness, provided that sheet tensile properties are approximately the same. Sheet ranging between 0.020 in. and 0.137 in. thickness were tested.

The metals industry has been without a commonly accepted test for toughness of sheet metal, that is, its resistance to withstand tearing in the plane of the sheet at high-strain rates. A variety of tests have been proposed which involve special equipment or instrumentation, present difficulties in testing over a range of temperatures or are otherwise inconvenient. This report describes the development and use of a specimen which will permit a quantitative measure of toughness by means of conventional pendulum-type impact testing apparatus. This specimen is fashioned by joining several blanks cut from the sheet to be tested to form a laminate from which the finished specimen may be machined. The laminae must be joined together in such a manner as to prevent twisting of the specimen during rupture (riveting may be employed satisfactorily). The specimen machined from the laminate corresponds to the conventional Charpy V-design except in width, which dimension is determined by the number of laminae selected so as to most nearly approach the standard value 0.394 in. (see Figs. 1 and 2). (For example, six laminae each

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[1] Chief, Physical Metallurgy Laboratory, Watertown Arsenal, Watertown, Mass.

0.070 in. thick would be joined to form a laminate 0.420 in. thick; all other multiples of 0.070 in. would deviate further from the 0.394 in. value.)

## PROCEDURE

In order to evaluate the behavior of laminated impact specimens without extraneous effects from processing variables associated with production of sheet in various gages, special specimens were fashioned from laminae machined from $\frac{1}{2}$-in. thick normalized, quenched-and-tempered low-alloy steel plate. The fine, homogeneous structure of this heat-treated plate is shown in Fig. 3. The laminae were surface ground from thin strips cut by a band saw parallel to the rolling direction of the plate. The various combinations of (*a*) lamina thickness, and (*b*) number of laminae per specimen are shown in Table I. Specimens comprising these various combinations were tested over a wide range of temperatures in order to determine the transition curve for each combination.

The transition temperature was defined as that at which 5 per cent of the fracture surfaces in the laminated specimen was brittle in appearance, an arbitrary criterion selected because of ease in definition. The effect of number of laminae and of aggregate thickness approximating 0.394 in. upon transition temperature is shown in Fig. 4. The transition temperature falls sharply as laminae increase from one to four, then decreases more slowly and linearly to eight, the maximum number investigated. Moreover, the minimum energy of the laminated specimens commensurate with 100 per cent ductile fracture decreased sharply, then more gradually with number of laminae. These results reflect the lessening of stress triaxiality in each individual lamina with decreasing thickness. The effect of aggregate thickness may also be observed.

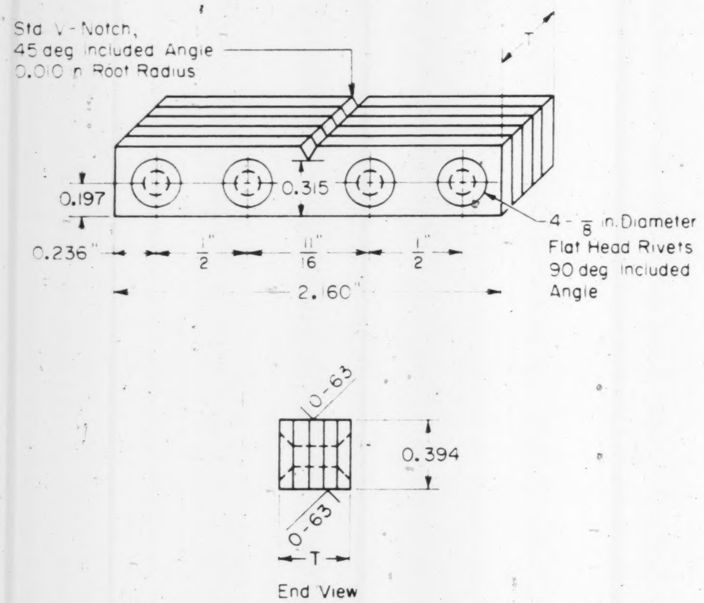

FIG. 1.—Laminated Specimen for Impact Testing of Sheet Metal.

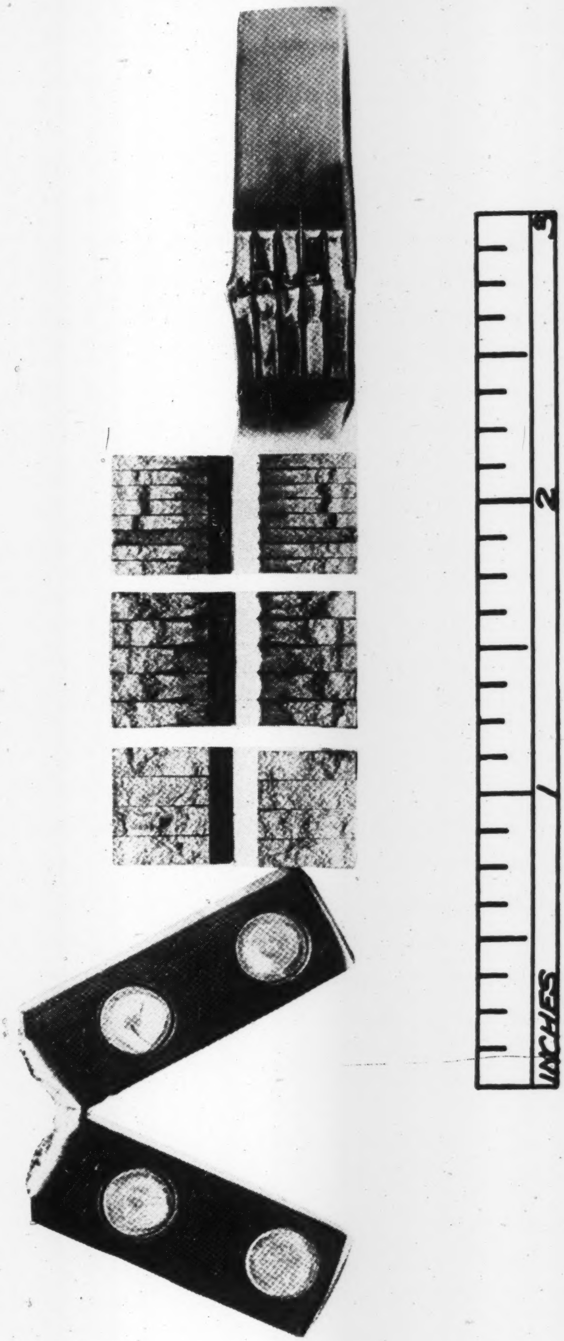

FIG. 2.—Laminated Impact Specimens for Testing Sheet Metal Toughness Showing Fracture Surfaces Commensurate with Various Degrees of Toughness.

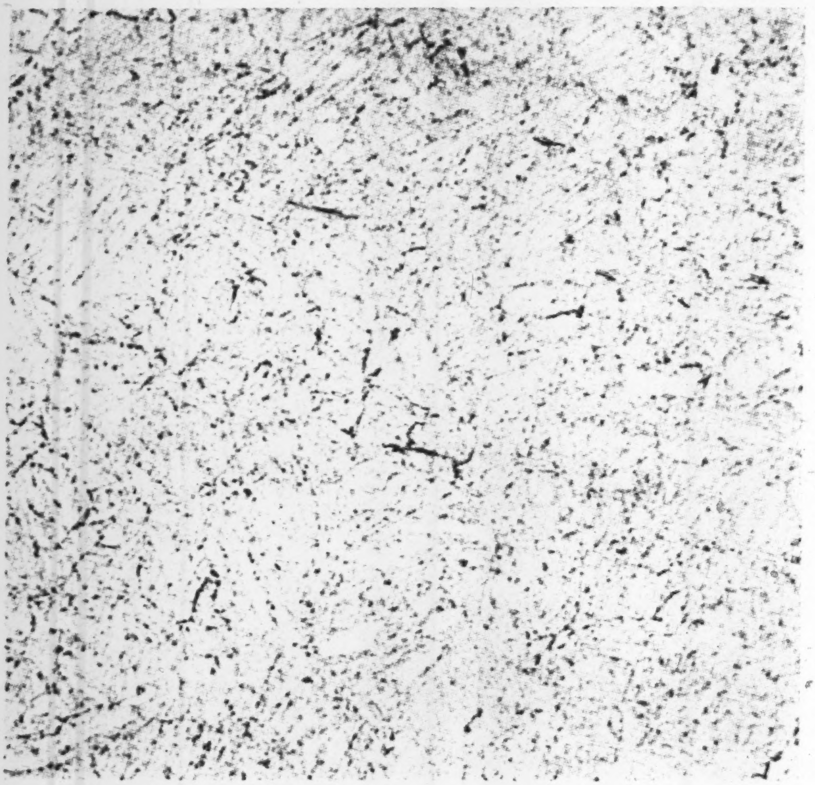

FIG. 3.—Representative Microstructure of Heat-Treated Low-Alloy High Tensile Steel Plate from which Laminae for this Investigation Were Fashioned, Nital Etch (× 1000).

Upon plotting the thickness of an individual lamina against (*a*) the transition temperature of the laminated impact specimen, and (*b*) the minimum energy of the individual lamina in ductile fracture, two important observations are apparent (see Fig. 5): both relations are remarkably linear over a range of thickness 0.050 to 0.200 in. (The linearity does not extend to the 0.400-in. thickness; thicknesses less than 0.050 in. were not tested in this series.)

Since sheet does not commonly exceed 0.25 in. in thickness, this behavior indicates the relation of sheet thickness to sheet metal toughness under this manner

TABLE I.—COMBINATIONS OF NUMBER AND THICKNESS OF LAMINAE TESTED IN DEVELOPMENT OF LAMINATED IMPACT SPECIMEN.

| Thickness of Laminae, in. | Number of Laminae | Width of Laminated Specimen, in. |
|---|---|---|
| 0.400 ± 0.005 | 1 | 0.400 |
| 0.200 ± 0.005 | 2 | 0.400 |
| 0.100 ± 0.005 | 4 | 0.400 |
| 0.090 ± 0.005 | 4 | 0.360 |
| 0.090 ± 0.005 | 5 | 0.450 |
| 0.080 ± 0.005 | 5 | 0.400 |
| 0.070 ± 0.005 | 5 | 0.350 |
| 0.070 ± 0.005 | 6 | 0.420 |
| 0.060 ± 0.005 | 6 | 0.360 |
| 0.060 ± 0.005 | 7 | 0.420 |
| 0.050 ± 0.005 | 8 | 0.400 |

of loading (a relation assuming identical mechanical properties for all thicknesses).

The value of these linear relationships may be anticipated in studies to determine the effect of processing or surfacing treatments on the toughness of sheet. In the tests reported above, considerable care was taken to achieve material homogeneity; treatments which alter this condition (that is, cold rolling, surface hardening, etc.) should cause deviation from the standard behavior. The relation between sheet thickness and minimum energy in ductile fracture is of importance since it indicates negligible resistance to failure under impact loading in low sheet thicknesses. This would appear to be the result of specimen geometry, since the material from which the special laminae were machined was inherently tough and should not have been embrittled by fashioning into specimens.

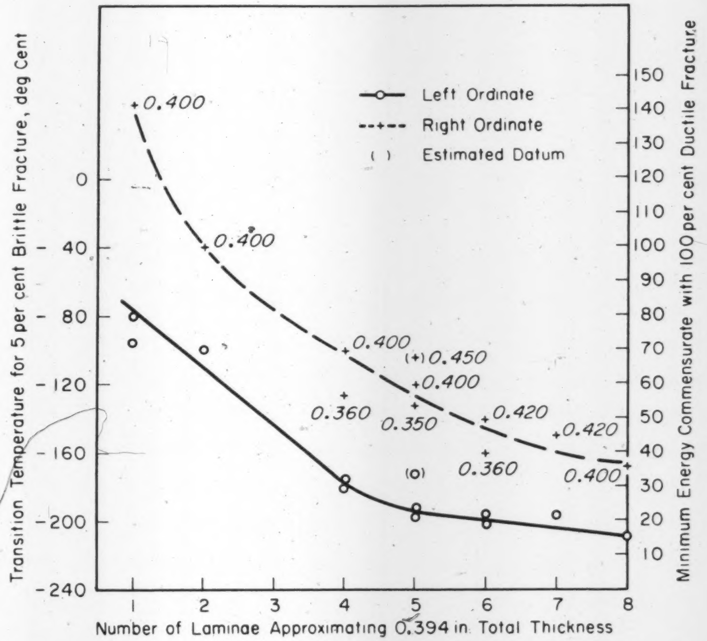

FIG. 4.—Transition Temperature for 5 per cent Brittle Fraction *and* Minimum Energy for 100 per cent Ductile Fracture *versus* Number of Laminae comprising Specimen.

## DISCUSSION OF SPECIMEN CHARACTERISTICS

In working with a new type of specimen, it is important to define its advantages and limitations. Reproducibility and sensitivity are necessary attributes. Sensitivity involves a variety of mechanical and metallurgical factors, only some of which have been studied to date.

Current interest in the use of titanium alloy sheet for aircraft and missile structures prompted investigation of the resistance of such material to tearing at high strain rates. The presently described laminated impact specimen was used in such an investigation. Since the sheet of various gages to each nominal composition differed somewhat in chemical composition, metallurgical history and surface condition, there was no reason to expect that the linear relationships observed for the carefully standardized steel specimens would be similarly demonstrated by titanium sheet. In some instances, differences in tensile ductility between sheet of one thickness to another, both being of the same composition, were reflected in deviations from the expected toughness: gage relationship. However, test data for sheet from several heats of the same nominal composition, titanium-6 per cent aluminum-4 per cent vanadium, in a range of gages from 0.020 to 0.125 in., provide a useful indication of behavior.

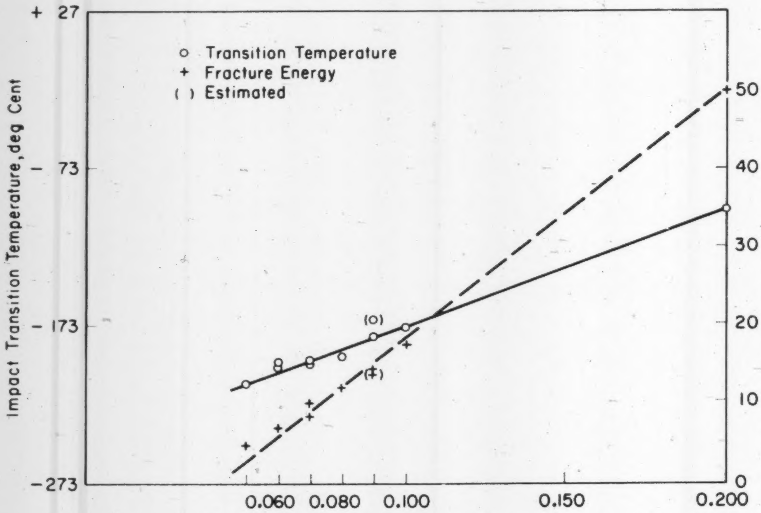

FIG. 5.—Thickness of Lamina in Inches.

The impact strengths of these laminated sheet specimens at various temperatures are plotted in Fig. 6 as a function of thickness. The toughness of the 0.078-in. sheet seems quite low in comparison with the apparent trend. In an effort to explain this deviation, these sheets were subjected to chemical analysis, metallographic examination, and tension testing (see Tables II and III). Since the path of fracture in the laminated specimen was always parallel to the rolling direction (by arbitrary sampling procedure), transverse tensile properties are particularly significant. It may be observed that the transverse yield and tensile strengths of the 0.078-in. sheet are appreciably higher than those of the 0.050 and 0.125-in. sheet, but are comparable with those of the 0.020-in. and lower than those of the 0.025-in. sheet. Whereas high transverse strength no doubt affects the toughness of the 0.020, 0.025, and 0.078-in. sheet to a similar

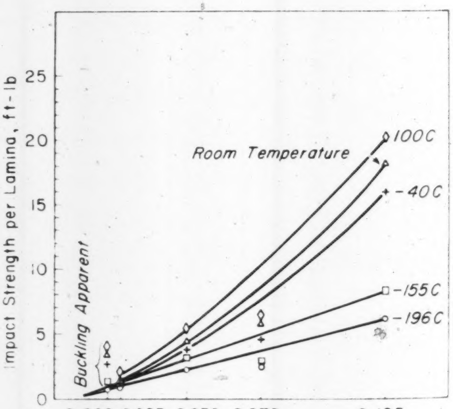

FIG. 6.—Toughness of Titanium Alloy Sheet (6 per cent Aluminum-4 per cent Vanadium).

extent on a fractional basis, the strength difference in foot-pounds will be negligible for the thinner material but significantly different for the 0.078-in. sheet. Accordingly, only the latter avoids the general relation, which apparently is linear. A similar relation between transverse strength and toughness was noted in tests of titanium-5 per cent aluminum-2.5 per cent tin alloy sheet from different heats but similar (0.047 and 0.050 in.) in gage. The transverse strengths of the 0.047-in. sheet were higher and its toughness lower. However, 0.040-in. sheet of similar high transverse strength, but low directionality, was as tough as the 0.050-in. sheet (with allowance for gage). It is apparent that transverse yield strength is not the only factor affecting toughness. Pertinent to this, the tensile and toughness properties of a titanium-8 per cent aluminum-2 per cent columbium-1 per cent tantalum sheet of 0.040 in. thickness (see Table III and Fig. 7) should be observed.

TABLE II.—ANALYSIS OF 6 PER CENT ALUMINUM, 4 PER CENT VANADIUM, TITANIUM ALLOY SHEET.

| Heat Number | Heat M5901 | | Heat M5900 | | Heat M5259 | | Heat M4947 | | Heat M4789 | |
|---|---|---|---|---|---|---|---|---|---|---|
| | TMCA[a] | WAL[b] | TMCA | WAL | TMCA | WAL | TMCA | WAL | TMCA | WAL |
| Aluminum... | 6.1 | 5.91 | 6.1 | 5.99 | 6.1 | 6.08 | 6.1 | 5.91 | 6.4 | 5.98 |
| Vanadium... | 4.0 | 4.04 | 4.2 | 3.39 | 4.1 | 3.93 | 4.0 | 3.53 | 3.9 | 3.75 |
| Iron........ | 0.08 | 0.14 | 0.08 | 0.12 | 0.11 | 0.15 | 0.09 | 0.16 | 0.10 | 0.14 |
| Carbon...... | 0.017 | 0.040 | 0.029 | 0.039 | 0.017 | 0.043 | 0.014 | 0.030 | 0.010 | 0.038 |
| Nitrogen.... | 0.009 | 0.013 | 0.008 | 0.014 | 0.013 | 0.013 | 0.012 | 0.011 | 0.010 | 0.012 |
| Hydrogen... | 0.015 | ... | 0.011 | ... | 0.008 | ... | 0.010 | ... | 0.006 | ... |

[a] Data reported by Titanium Metals Corp. of America.

[b] Data reported by Watertown Arsenal Laboratories.

In the limited work completed to date, no correlation of toughness with base composition has been possible. Likewise, surface contamination and orientation effects have not been explored.

On occasion, the laminated specimen has not behaved satisfactorily in testing sheet less than 0.025 in. in thickness, particularly when the material was tough in nature. In these instances, the laminae buckled in process of rupture, thereby expending energy in deformation unassociated with tearing. Minor distortion will not affect the datum appreciably, but some sheet has proven so tough as to buckle severely with no initiation of rupture at base of the notch whatsoever. This behavior can sometimes be avoided by testing at lower temperatures. In the case of 0.020-in. sheet of the alloy titanium-6 per cent aluminum-4 per cent vanadium, buckling persisted to −155 C; but at −196 C, the next lower testing temperature, no buckling was observed and the datum fell nicely into line with those of heavier gages tested at this same temperature (see Fig. 6).

Since rupture produces some lateral expansion of the individual laminae in the vicinity of tup contact, this may cause the two inner rivets to stretch very slightly, thereby expending energy unassociated with tearing. However, tests of specimens from which these two inner rivets were removed (after assembly and machining) showed no deviation from data obtained with the standard specimens. (The four-rivet design remains preferred since it prevents separation of the laminae during machining of the V-notch with a fly cutter.)

Aside from the readily detectable tendency for buckling in specimens of thin sheet, the laminated specimen has

TABLE III.—TENSILE PROPERTIES OF TITANIUM ALLOY SHEET.

| Alloy, Heat Number | Gage, in. | Direction | Yield Strength, psi[a] | Tensile Strength, psi | Elongation, per cent[b] |
|---|---|---|---|---|---|
| TITANIUM-6 PER CENT ALUMINUM-4 PER CENT VANADIUM | | | | | |
| M5901 | 0.020 | longitudinal | 130 000 | 131 000 | 13 |
| | | longitudinal | 139 000 | 140 000 | 12 |
| | | 45 deg | 138 000 | 140 000 | 10 |
| | | 45 deg | 141 000 | 141 000 | 10.5 |
| | | transverse | 139 000 | 140 000 | 9.5 |
| | | transverse | 139 000 | 139 000 | 11.0 |
| M5900 | 0.025 | longitudinal | 139 200 | 145 400 | 13.0 |
| | | longitudinal | 143 100 | 143 800 | 13.0 |
| | | 45 deg | 137 700 | 138 500 | 12.5 |
| | | 45 deg | 138 800 | 139 200 | 12.5 |
| | | transverse | 149 200 | 150 000 | 15.0 |
| | | transverse | 147 700 | 149 200 | 14.0 |
| M5259 | 0.050 | longitudinal | 107 000 | 110 500 | 15.0 |
| | | longitudinal | 115 000 | 120 000 | ... |
| | | 45 deg | 117 000 | 117 000 | ... |
| | | 45 deg | 117 000 | 117 000 | 15.5 |
| | | transverse | 123 300 | 123 300 | 12.0 |
| | | transverse | 125 000 | 125 000 | ... |
| M4947 | 0.078 | longitudinal | 130 500 | 132 000 | 16.5 |
| | | longitudinal | 132 500 | 140 000 | ... |
| | | 45 deg | 129 500 | 130 000 | ... |
| | | 45 deg | 130 000 | 130 000 | 16.0 |
| | | transverse | 140 000 | 141 000 | 14.0 |
| | | transverse | 142 500 | 143 250 | 15.0 |
| M4789 | 0.125 | longitudinal | 121 400 | 129 200 | ... |
| | | longitudinal | 123 800 | 132 100 | 17.0 |
| | | 45 deg | 125 100 | 125 400 | 19.0 |
| | | 45 deg | 125 100 | 125 400 | 16.5 |
| | | transverse | 131 700 | 131 700 | 15.0 |
| | | transverse | 130 200 | 131 700 | ... |
| TITANIUM-8 PER CENT ALUMINUM-2 PER CENT COLUMBIUM-1 PER CENT TANTALUM | | | | | |
| MST 25725 | 0.040 | longitudinal | 128 400 | 128 900 | 25.0 |
| | | longitudinal | 128 900 | 128 900 | 25.0 |
| | | 45 deg | 128 900 | 131 600 | 24.5 |
| | | 45 deg | 131 100 | 131 600 | 23.0 |
| | | transverse | 130 600 | 131 100 | 28.0 |
| | | transverse | 130 600 | 130 600 | 27.0 |
| TITANIUM-5 PER CENT ALUMINUM-2.5 PER CENT TIN | | | | | |
| D1-214530 | 0.040 | longitudinal | 134 200 | 134 700 | 17.0 |
| | | longitudinal | 136 300 | 136 800 | 19.0 |
| | | 45 deg | 136 700 | 137 200 | 20.5 |
| | | 45 deg | 133 900 | 134 400 | 19.0 |
| | | transverse | 133 800 | 135 300 | 20.0 |
| | | transverse | 131 800 | 132 900 | 19.0 |
| 41208 | 0.047 | longitudinal | 123 900 | 137 000 | 19.0 |
| | | 45 deg | 127 800 | 127 800 | 18.5 |
| | | transverse | 135 700 | 136 100 | 19.5 |
| 43180 | 0.050 | longitudinal | 119 200 | 120 000 | 15.0 |
| | | 45 deg | 122 000 | 124 000 | 16.5 |
| | | transverse | 128 800 | 128 800 | 20.0 |
| D30540 | 0.137 | longitudinal | 120 600 | 122 500 | 20.0 |
| | | transverse | 119 100 | 121 200 | 21.0 |

[a] Yield strength at 0.2 per cent offset. [b] Per cent elongation in 2 in. gage length.

proven quite reproducible. Some triplicate data demonstrating this quality are shown below:

IMPACT STRENGTH OF LAMINATED SPECIMENS FASHIONED FROM 0.133 IN. SHEET OF ALLOY TITANIUM WITH 8 PER CENT MANGANESE.

| −40 C | 100 C | 300 C |
|---|---|---|
| 3.0 | 7.5 | 16.0 |
| 3.0 | 7.5 | 16.0 |
| 3.0 | 7.0 | 16.5 |

Because the specimen reports the aggregate energy absorption of several laminae, the effect of a local inhomogeneity in the sheet is alleviated; the average value obtained by dividing the aggregate energy to rupture by the number of laminae is therefore more nearly representative than test of a single sheet specimen.

## CONCLUSIONS

The laminated specimen for Charpy impact testing of sheet metal has been shown to yield reproducible test data. It is also sensitive to the effect of metallurgical variables upon sheet toughness.

Test of specimens fashioned from laminae machined from steel plate disclosed linear relations for (*a*) the impact transition temperature and (*b*) the minimum energy of an individual lamina in ductile fracture *versus* thickness of an individual lamina. This linearity maintained over a range of thickness from 0.050 in. to 0.200 in.

Test of laminated specimens prepared from titanium alloy sheet of various compositions indicated that impact strength for a given composition at a selected temperature is closely proportional to sheet thickness, providing that sheet tensile properties are approximately the same. Sheets ranging between 0.020 in. and 0.137 in. in thickness were tested.

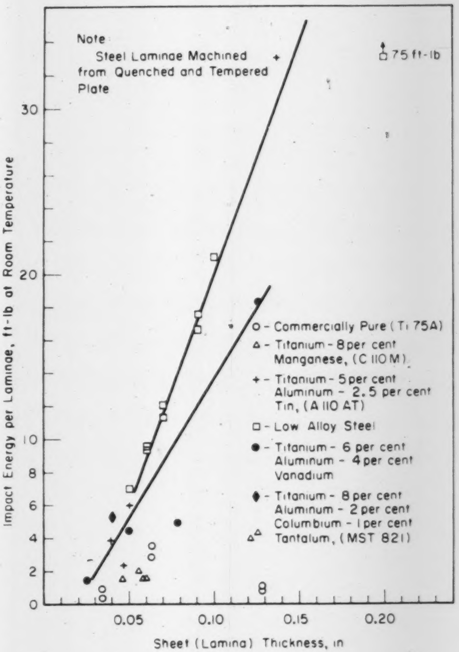

FIG. 7.—Toughness *versus* Thickness of Titanium-Alloy Sheet and Low-Alloy Steel Sheet.

# AN EVALUATION OF GEIGER COUNTER X-RAY TECHNIQUES FOR MEASURING STRESSES IN HARDENED STEELS*

By Karl E. Beu[1]

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## Synopsis

A "two exposure" X-ray Geiger counter technique for measuring residual and applied stresses is discussed. This technique was originally described by Christenson and Rowland (**4**)[2]. A comparison of results published by other observers using similar techniques is also discussed and evaluated. Experimental and theoretical evidence is offered to demonstrate that the method described by Christenson and Rowland (**4**) is useful for making absolute stress measurements with an accuracy of about ±10,000 psi[3] and a reproducibility of about ±5000 psi[3] on steels in the hardness range of 25 to 62 Rockwell C. To date, this method has been found to be the only reliable one for measuring stresses in hardened steels by X-rays.

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Historical Note.—In the Research Conference on Residual Stress Problems,[4] a symposium was presented on X-ray methods for measuring residual stresses in which the merits of various Geiger counter techniques were discussed. This discussion, in addition to recent publications, resulted in a considerable confusion as to the merits of the various X-ray methods. This paper is based on a careful, objective survey and on experimental work using the various methods reported in an attempt to resolve the differences and also to emphasize the validity of the recommended method for stress measurements when used within its limitations.

The experimental work discussed in this paper was carried out by General Motors' research staff personnel. Because of General Motors publication policies, permission to publish this paper has been granted only recently. In spite of the time interval between the Sagamore Conference and now, it is the belief of the author that this paper still merits publication, since to his knowledge, no paper has appeared in the literature which discusses the relative merits of the various Geiger counter X-ray techniques for measuring residual stresses in hardened steels.

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The use of X-ray diffraction techniques for measuring residual stresses[5] has been discussed for over twenty years in the literature. It has been consistently pointed out that the diffuseness of diffraction rings or peaks from hardened steel makes it difficult, if not impossible, to measure residual stresses in such material. For example, it has been stated that stresses cannot be measured in steels that have been hardened above 40 Rockwell C (1) or, more recently, "The hardness limitation for acceptable measurements is in the neighborhood of 45 $R_c$" (2).

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1290.

[3] Deviations given here and below are average deviations.

[4] Sponsored by Syracuse University Research Inst. and the Ordnance Corps of Watertown Arsenal held at the Sagamore Conference Center, Sagamore, N. Y., Aug. 19–20, 1954.

[5] Measurements referred to in this paper are to macro-stresses (stresses of the same sign in relatively large groups of grains in one sample and which cause diffraction line shift) as contrasted to micro-stresses (non-uniform stresses within individual grains which contribute to diffraction line broadening).

Only recently has a two exposure X-ray method (3) been suggested by Christenson and Rowland (4) which makes it possible to make residual stress measurements in steels as hard as 60 Rockwell C. The experimental technique used for this method involves the use of a Geiger counter diffraction unit with chromium K$\alpha$ radiation and a 0.001 in thick vanadium metal foil filter in front of the Geiger tube window. Scaled diffraction data are obtained of the martensite (211) diffraction peak at two different angles of sample inclination to the primary X-ray beam. The straight-line portions of the sides[6] of the (211) peak are algebraically extrapolated to their point of intersection, this intersection point being defined as the "peak position" in terms of degrees $2\theta$.[7] The difference in diffraction angle of the peak positions for two exposures is then related directly to stress parallel to the surface of the hardened steel sample after a special correction has been made based on the slopes of the sides of the (211) diffraction peak. It has been found that this particular technique is very satisfactory, not only for obtaining accurate and reproducible stress measurements on 60 Rockwell C steels, but also for steels in the hardness range of about 30 to 60 Rockwell C.

## DISCUSSION OF THE CHRISTENSON AND ROWLAND METHOD

In using elastic theory to relate interplanar spacings to applied or residual stress, it is necessary to make some assumptions; (*a*) The strain in the sample grains or crystallites contributing to the diffraction effect is representative of the strain in the sample surface. (*b*) The values for Young's modulus E and Poisson's ratio $\nu$ can be calculated for each group of grains contributing to the diffraction effect (see Appendix I for value of symbols). Christenson and Rowland assume that (*a*) is true and demonstrated experimentally that in (*b*) the engineering values for E and $\nu$ can be used for the (211) planes in hardened steel (alpha iron) regardless of their orientation with respect to the applied or residual stress.

The validity of these assumptions has been verified experimentally for hardened steels as discussed below, and may be related to the relatively large number of grains of alpha iron (martensite) irradiated by the primary X-ray beam which contribute to the diffraction effect. The size of the martensite needles in hardened steel is very small compared to the size of ferrite grains in well-annealed steel or other soft metals. If the number of grains irradiated by the X-ray beam is very large, it may be that those which contribute to the diffraction effect at a particular Bragg angle for various angles of incidence of the X-ray beam are substantially representative of the sample. Furthermore, it may be that the very smallness of the martensite grains in hardened steels may restrict plastic flow within the grains such as is encountered in large grained metals and which has been reported to give anomalous stress results for various soft metals such as annealed steel. In order to demonstrate the validity of these assumptions and ideas, it may be worthwhile to review the basic stress equation applicable to this problem. This equation is given in two forms as follows:

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[6] Straight line portions for the sides of the martensite (211) peak have been observer within the experimental error of measurement with little or no ambiguity among a group of five observers. This was done for enough calculations to demonstrate the validity of the existence of straight-line portions.

[7] It is more correct to define an axis as the peak position. The axis in this case is the bisector of the line side slopes. This will be discussed in more detail in a later section.

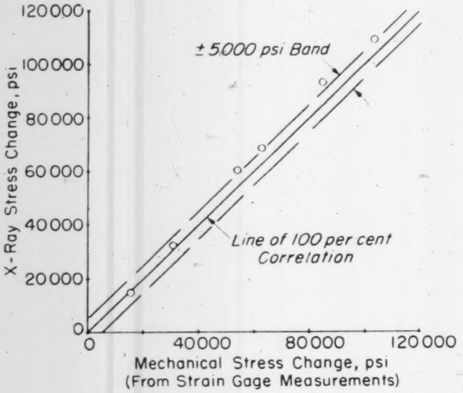

FIG. 1.—Correlation of X-ray and Strain Gage Stress Measurements for SAE 1095 Steel Hardened to 60 Rockwell C.

$$S = \frac{E}{d_0(1+\nu)} \cdot \frac{1}{\sin^2\psi} \cdot (d\psi - d_{\parallel})$$

$$= K(d\psi - d_{\parallel}) = K\Delta d \quad (1)$$

$$d\psi = \left[\frac{d_0(1+\nu)}{E} S\right] \sin^2\psi + d_0\left(1 - \frac{\nu S}{E}\right)^* \quad (2)$$

See appendix for symbols.

The stress equation as given in Eq 2 is identical to that of a straight line, namely, $y = mx + b$, where the variables are $d\psi$ and $\sin^2\psi$. A plot of the stress equation in Eq 2 has the following characteristics: (*a*) for $d\psi = d_0$, $\sin^2\psi = \frac{\nu}{1+\nu} = 0.23$ if engineering values are used for E and $\nu$, and (*b*) the slope of the straight line is proportional to the stress. If these characteristics can be observed experimentally for hardened steel, it would indicate that the Christenson and Rowland calculation procedure, arbitrary though it may seem, is on a firm experimental and theoretical footing from the viewpoint of elastic theory.

These characteristics have been observed for a sample of SAE 1095 steel hardened to 60 Rockwell C. In addition, excellent correlations have been obtained with strain gage measurements over a range of about 100,000 psi applied stress for this sample.[8] The correlation measurements which are directly related to Eq 1 are indicated in Fig. 1. A plot of Eq 2 taken from the same set of data used in Fig. 1 is given in Fig. 2. As can be seen in Fig. 1, the X-ray and strain gage measurements agree within about ±5000 psi from zero to 109,000 psi applied stress. In Fig. 2 the $d\psi$ *versus* $\sin^2\psi$ experimental points for a given stress level all fall on straight lines within experimental error. These straight lines intersect very closely at $\sin^2\psi = 0.23$

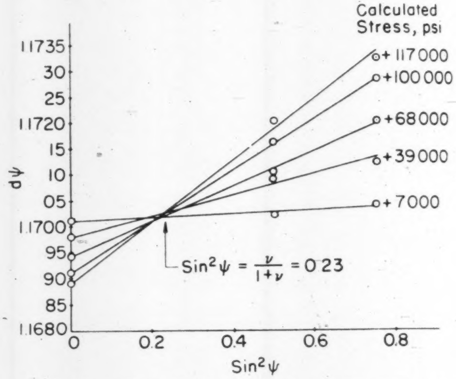

FIG. 2.—Plot of $d\psi$ *versus* $\sin^2\psi$ for a sample of SAE 1095 Hardened Steel Mechanically Stressed in the Range of about 0 to 120,000 psi.

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* This expression is only valid for a unidirectional surface stress and in event of bi-directional stress, it is necessary to substitute the quantity $d_0\,[1 - (\nu S'/E)]$, where $S'$ is the stress at right angle to $S$, for $d_0$ in the equation.

[8] A sample loading fixture similar to that of Bennett and Vacher (see Fig. 4 of Reference **14**) was built for applying tensile stresses to a thin flat specimen. An SR-4 strain gage was fastened to the opposite side of the steel strip from which X-ray measurements were made.

as predicted by elastic theory and using the engineering values for Young's modulus and Poisson's ratio. Finally, the ratios of slopes vary directly as ratios of calculated stresses.

It has also been observed experimentally that the calculated stress is essentially zero on the surfaces of hardened steel samples which are believed to be stress free. Such samples include (*a*) steel hardened by cooling so slowly below the Ms transformation temperature that it has no macro-stresses;[9] (*b*) hardened steel from which metal has been removed until the remaining calculated stress is essentially zero according to mechanical measurements using dissectioning techniques (**7**) (additional metal was taken off electrolytically before X-ray measurements were made to remove all traces of the superficial stresses induced by the mechanical metal removal program); and (*c*) hardened steel powder in which macro-stresses are essentially absent because of the small particle size of the powder.

Finally, excellent correlation has been obtained between X-ray stress measurements and dissectioning-type stress measurements (**7**) with depth through the case of a carburized bar and covering a hardness range of 25 to 62 Rockwell C. This indicates that the Christenson and Rowland method is valid for at least one steel over a wide range of hardnesses. These experiments all indicate that the engineering and X-ray stress constants agree, using the Christenson and Rowland method, and that absolute values of stress calculations are accurate to about ±10,000 psi, with a reproducibility of about ±5000 psi. Experimental work correlating X-ray and mechanical stress measurements (**7**) indicate that the accuracy may be better than this, or about ±5000 psi. This is based on obtaining numerous results by both methods which do not disagree by more than about 10,000 psi.

## EVALUATION OF AND COMPARISON WITH SIMILAR METHODS

Various evaluations have been made in the past several years of the validity of X-ray stress measurements both on annealed and hardened steels using several different criteria for measuring peak positions. It has been emphasized that

> "The bulk modulus value of $30 \times 10^6$ psi and Poisson's ratio of 0.28 used together in the term $\frac{E}{1+\nu}$ are valid for chromium radiation for both the austenite and martensite phases at all $\psi$ orientations to the direction of stress" (**4**).

However, it has also been pointed out that

> "The value of the stress constant [involving E and $\nu$] is 32 per cent higher [for the martensitic phase in hardened steel using chromium radiation] than the theoretical value" (**9**).

Obviously, these points of view are contradictory. In an attempt to resolve some of these apparent discrepancies, the following ideas are summarized:

For mild or annealed metals, including steel, in which the grain size is relatively large, the anisotropic response of groups of grains to applied stress is well known (**5, 6**). On this basis, variations between X-ray and engineering values for E and $\nu$ may be expected for mild steel. Along this line, Maloof (**8**) and Letner and Maloof (**9**) have stated that, for annealed SAE 8742 and annealed tool steel, the X-ray value for E is 30 to 40 per cent higher than the engineering value using chromium radiation and the (211) planes of iron. Ogilvie (**10**) has mentioned, however, that the X-ray and engineering values for E and $\nu$ seem to agree when using cobalt radiation and the (310) planes of iron. Hence, it seems that the stresses calculated using the (310) planes are representative of a sample of *annealed* steel while those calculated using the (211) planes are not representative. On the other hand, for *hardened* steel, Ogilvie (**10**) and Blount and Ellis in their discussion of the Christenson and Rowland (**4**) agree that the X-ray values of the stress constant are 20 to 30 per cent higher than the engineering values while Christenson and Rowland (**4**) and the author find that the X-ray and engineering values of the stress constant agree within experimental error. These several results have been summarized in Table I. The problems due to anisotropic response have been recognized and no solution for them is offered; however, the discrepancies noted in Table I for hardened steels may be resolved by a consideration of the absorption correction factor discussed by Christenson and Rowland (**4**).

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[9] This sample was kindly supplied by A. L. Christenson, Timken Roller Bearing Co., Canton, Ohio, and is a block 1 by ⅞ by ⅝ in. of modified 52100 steel (1 per cent carbon, 0.8 per cent manganese, 1.5 per cent chromium, 0.2 per cent nickel, 0.3 per cent molybdenum) heat treated as follows: austenitized 10 min at 1550 F, quenched in salt to 500 F (about 20 F above Ms), held 1 min, cooled very slowly in Sil-o-Cel to room temperature, tempered 2 hr at 350 F.

TABLE I.—COMPARISON OF X-RAY STRESS DATA FROM EIGHT DIFFERENT OBSERVERS.

| Observer | Diffraction Peak Used | Characteristic Kα Radiation Used | Type of Peak Measurement Used[a] | Type of Steel | Rockwell C Hardness | Agreement of X-ray Stress Constant $K$ with Engineering Values |
|---|---|---|---|---|---|---|
| Maloof (**8**) | (211) | Chromium | Centerline[b] | Annealed SAE 8742 | Not given | 30 to 40 per cent higher |
| Letner and Maloof (**9**) | (211) | Chromium | Centerline[b] | Annealed tool steel | Not given | 30 per cent higher |
| Ogilvie (**10**) | (211) | Chromium | Centerline[b] | Annealed SAE 52100 | Not given | 22 per cent higher |
| Ogilvie (**10**) | (211) | Chromium | Parabolic (**10**) | Hardened SAE 52100 | 65 | 32 per cent higher |
| Ogilvie (**10**) | (310) | Cobalt | Centerline[b] | Annealed 52100 | Not given | Agrees within experimental error |
| Blount and Ellis discussion of the paper by Christenson and Rowland (**4**) | (211) | Chromium | Parabolic (**10**) | Carburized and hardened AISI 4720 | 62 | 20 per cent higher |
| Christenson and Rowland (**4**) | (211) | Chromium | Line slope (**4**) | Carburized and hardened SAE 4620 | 60+ | Agrees within experimental error |
| Beu | (211) | Chromium | Line slope (**4**) | Hardened SAE 1095, carburized and hardened SAE 9310 | 30 to 62 | Agrees within experimental error |

[a] All observers used the two exposure method.

[b] Position of the intersection of a centerline with the experimental diffraction peak curve. This centerline is obtained by drawing a line through a series of points located midway between the sides of the diffraction peak curve.

Using the two exposure method, this correction factor can be shown to be due primarily to differences in the absorption of X-rays due to different X-ray path lengths into and out of the sample surface for various $\psi$ angles. This absorption effect has been previously discussed by Wilchinsky (**11**). This correction factor (but *not* the anistropy problem) may be ignored for mild steel where the $\alpha_1\alpha_2$ doublet is resolved and the angular width of the diffraction peaks is very small. It cannot be ignored, however, when working with very broad diffraction peaks such as those obtained from hardened steel. Furthermore, since this correction factor is multiplicative in nature, applying to the diffracted as well as the background intensity, corrections cannot be made merely by subtracting background scattering from diffraction peak intensity measurements. Neither can this correction factor be obtained directly from the background scattering curve since it is experimentally impossible, in general, to obtain the background curve on the high angle side of the martensite (211) diffraction peak. The correction, therefore, is made by algebraically adjusting the slopes of the straight-line portions of the martensite (211) peak to symmetry with respect to the abscissa (degrees $2\theta$) for each of two $\psi$ angles.

By studying the implications of this absorption correction, it can be demonstrated that the arbitrariness inherent in the Christenson and Rowland method essentially cancels out by using the two-exposure technique as part of this method. This is not true if single-exposure techniques are used, since the Christenson and Rowland definition of peak position for an individual measurement as given previously is entirely arbitrary; however, it should be made clear that any definition of peak position would, in general, be arbitrary for broad diffraction peaks. For single exposure measurements, the results depend, among other things, on the availability and use of hardened steel standard stress samples. To date, these have not been available for X-ray measurements except those which have been calibrated using the Christenson and Rowland method.

A definition of peak position implies the definition of an axis of that peak. Such axes have already been indicated and include the following: (*a*) the bisector of the angle formed by the straight-line portions of sides of a diffraction peak (**4**), (*b*) a center line as defined in the footnote of Table I (**8, 9**), and (*c*) the axis of a parabola which has been fitted by least squares in the vicinity of the maximum of a diffraction peak. Stress is then calculated from a measurement of the distance between two axes obtained at two different $\psi$ angles. Since it is an experimentally observed fact that the axes for two different $\psi$ angles are not parallel according to definitions (*a*) or (*b*) above, it is necessary to bring them into parallelism by a suitable mathematical manipulation before the distance between them can be measured.

Parallelism of "peak position" axes for two $\psi$ angle measurements is accomplished with the Christenson and Rowland method by applying the absorption correction. In practice this is done by algebraically rotating the martensite (211) peak until the slopes of the sides of the peak are symmetrical with respect to the abscissa (degrees $2\theta$) as pointed out above. The bisector of the angle between these line slopes will then be perpendicular to the abscissa. Rotation of peaks for two $\psi$ angles according to this criterion will cause both bisectors to be perpendicular to the abscissa and, hence, parallel to each other. The axis of rotation is immaterial just so that it is the same for both $\psi$ angle measurements. Whether or not the peaks have a true axis of symmetry is also immaterial since the bisector of the line side slopes is arbitrarily chosen as this axis. It has been found experimentally that the artificial symmetry introduced by rotating the peak, so that this bisector is perpendicular to the abscissa, does not affect the accuracy of the final stress results.

The use of other methods for hardened steels (8, 10, 12) without making an absorption correction of the type discussed above may result in misleading stress calculations when using the two exposure technique. For example, if a parabola with an axis perpendicular to the abscissa is fitted to a diffraction peak (10) with an inclined axis,[10] the peak position of this parabola may well differ from that of a parabola fitted to a diffraction peak which has been previously rotated algebraically according to the symmetry criterion of Christenson and Rowland. It is possible that the discrepancy of 0.836 deg noted by Blount, Thourson, and Ellis (13) between two measurements at $\psi$ angles of 0 deg and 45 deg in a macro-stress-free sample is due to trying to fit a parabola with a vertical axis to a diffraction peak with an inclined axis. This difference of 0.836 deg corresponds to a stress change of about 75,000 psi if engineering values are used for E and $\nu$. Some preliminary experimental work indicates that, if a parabola is fitted to a rotated peak, the stress results obtained with this modified form of Ogilvie's method (10) agree with the results obtained using the Christenson and Rowland method.

It has also been found that other factors such as the need for (*a*) resolving the $\alpha_1\alpha_2$ doublet for a broadened peak (12), (*b*) resolving the tetragonal martensite doublets (except perhaps for the case of completely untempered hardened steel which is of little practical importance), and (*c*) correcting for peak skewness due to rapidly changing diffraction dispersion or X-ray beam geometry are of secondary importance compared to the absorption correction factor for hardened steels.

## CONCLUSIONS

The Christenson and Rowland method has proven to be the only method to date for obtaining agreement between the engineering and X-ray stress constants. It is believed that this agreement is not fortuitous but rather is due to Christenson and Rowland having (*a*) recognized the need for an absorption correction, (*b*) made this correction in an arbitrary but consistent manner, and (*c*) used this correction in connection with the two exposure technique so that the factor of arbitrariness cancels out. It is worthwhile emphasizing the fact that the Christenson and Rowland method gives consistent results for macro-stress-free samples, for samples stressed elastically, and for samples containing residual stresses. No other method has been found to date which can be used this generally for hardened steel samples. Finally, experimental work has been done which indicates that the application of the Christenson and Rowland method provides absolute stress measurements on hardened steels accurate to about ±10,000 psi with a reproducibility of about ±5000 psi.

*Acknowledgments:*

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[10] Peak axis may be defined here, for example, as the line side slope bisector.

## REFERENCES

(1) W. T. Sproull, "X-Rays in Practice," 1st Ed., McGraw-Hill Book Co., New York, N. Y., p. 469 (1946).

(2) C. R. "Lewis, Stress Analysis by X-ray Diffraction," *Nondestructive Testing*, Vol. 8, pp. 18-22 (1949).

(3) C. S. Barrett, "Structure of Metals," 1st Ed., McGraw-Hill Book Co., New York, N. Y., pp. 273-275 (1943).

(4) A. L. Christenson and E. S. Rowland, "X-ray Measurement of Residual Stress in Hardened High Carbon Steel," *Transactions*, Am. Soc. Metals, Vol. 45, pp. 638-676 (1953).

(5) G. B. Greenough, "Residual Lattice Strains in Plastically Deformed Polycrystalline Metal Aggregates," *Proceedings*, Royal Soc., Vol. 197A, pp. 556-567 (1949).

(6) R. I. Garrod, "Residual Lattice Strains in Mild Steel," *Nature*, Vol. 165, pp. 241-242 (1950).

(7) M. Miller, E. Mantel, and W. S. Coleman, Jr., "An Evaluation of the X-ray Diffraction Method of Stress Measurement with a Comparison to Dissection Methods of Residual Stress Measurement in Hardened Steel," *Paper No. 411*, Annual Meeting, Soc. Experimental Stress Analysis, Columbus, Ohio, Oct. 31-Nov. 2, 1956.

(8) S. R. Maloof, "X-ray Strain Measurement Techniques with a Norelco Wide-Range X-ray Diffractometer," *Norelco Reporter*, Vol. 1, pp. 35-39 (1954).

(9) H. R. Letner and S. R. Maloof, "Stress Measurements by X-ray Diffraction," *Journal of Applied Physics*, Vol. 25, p. 1440 (1954).

(10) R. Ogilvie, "Stress Measurements with the X-Ray Spectrometer," *M.S. Thesis*, Massachusetts Institute of Technology (1952).

(11) Z. Wilchinsky, "X-ray Measurement of Order in the Alloy $Cu_3Au$," *Journal of Applied Physics*, Vol. 15, pp. 806-812 (1944).

(12) L. G. Finch, "A Correction to the Diameter Measurement of Diffuse X-ray Diffraction Rings," *Nature*, Vol. 163, pp. 402-403 (1949).

(13) E. I. Blount, T. L. Thourson, and A. L. Ellis, "Recent Developments in X-ray Stress Analysis," Paper presented at the December, 1953, Meeting of the Society for Experimental Stress Analysis.

(14) J. A. Bennett and H. C. Vacher, "Calibration of X-ray Measurement of Strain," *Journal of Research*, National Bureau of Standards, Vol. 40, pp. 285-293 (1948).

## APPENDIX I

## SYMBOLS

$S$ = component of stress parallel to the sample surface, the direction of which is in the plane formed by the incident and diffracted X-ray beams.

$\psi$ = angle between the diffracting planes and the stress direction. Since this paper is concerned primarily with the (211) planes of alpha iron, this and following symbols will be defined for the special case of the (211) planes of alpha iron (martensite) when using chromium $K\alpha$ radiation.

$d\psi$ = interplanar spacing of grains in which the (211) planes are inclined at $\psi$ degrees to the stress direction and the normals of the (211) planes lie in the plane of the incident and diffracted X-ray beams.

$d_{\parallel}$ = interplanar spacing in which the (211) planes are parallel to the stress direction and the sample surface.

$d_0$ = stress-free interplanar spacing of (211) planes for which either $d_{\parallel}$ or $d\psi$ may be substituted with negligible error.

$K$ = stress constant.

$E$ = Young's modulus = $30 \times 10^6$ for steel (engineering values) see page 660 of reference (4).

$\nu$ = Poisson's ratio = 0.28 for steel (engineering values) see p. 660 of reference (4).

# NONDESTRUCTIVE TECHNIQUES FOR MACRO- AND MICROGRAPHIC SURFACE EXAMINATION OF METALLIC SPECIMENS (ELECTROLYTIC LOCAL POLISHING AND REPLICA TECHNIQUE)*

By Pierre-A. Jacquet[1]

Various methods of making replicas for examination of the structure of metals and alloys with the electron microscope have seldom been applied in conventional metallography. The literature contains only a very small number of examples indicating that study of a replica can present certain advantages in comparison with the study of the metallic surface itself. Two cases described in detail the application of the Faxfilm method (**1**)[2] to fatigue testing (**2**) and to strongly radioactive materials (**3**).

It is obvious that the taking of replicas on the entire surface of a part being tested, at successive stages of fatigue, permits the making of records by which one can easily follow the changes in the surface, which is very difficult if not impossible to do with a test specimen. In the case of materials of high radioactivity a replica obtained with the help of apparatus controlled from a distance could be studied without special precautions.

Except for these particular cases the substitution of a replica for the object itself does not present advantages sufficiently convincing to justify the additional difficulty. But it is different when the metallic structure of parts whose form and shape do not permit direct examination under the metallographic microscope, and parts which must remain intact, are to be studied. It is surprising that replica techniques have not been developed in the field of nondestructive macro and micrographic examinations, since destruction of the part, necessary for the taking of a small specimen, would be obviated. Another more important advantage is that transparent replicas can be examined in an ordinary microscope and, even for macrography, with simple optical apparatus. Thus metallography would be possible in any studio or laboratory not equipped with the costly apparatus that has been considered indispensable in the observation of the surface of opaque bodies.

The general use of replicas in areas where they are particularly desirable, that is, in nondestructive examinations, requires (1) a simple rapid method giving a faithful replica of a surface of any shape or size; (2) a preparatory technique adequate for the surface under consideration.

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Translated from the French by Beatrice Marvin of the ASTM Editorial Staff.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1302.

The present paper proposes solutions to both of these problems.

## TECHNIQUE OF LOCAL POLISHING

### *General Considerations:*

To obtain a high degree of polish on a limited section of a large part, the exclusive use of abrasives and polishing preparations requires time, and often presents great difficulties. One therefore immediately thinks of replacement of mechanical polishing by an electrolytic process such as that used for a long time for small specimens (**4**). A method of local electrolytic polishing has been proposed by Mazia (**5**); its form is however much too simplified to be applied successfully in actual practice.

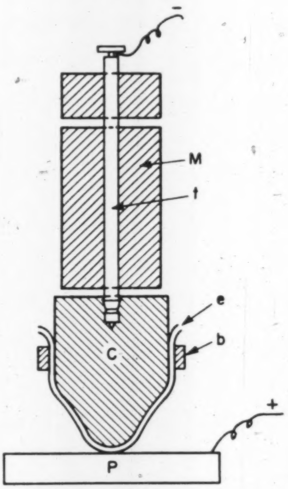

FIG. 1.—Diagram of "Plug" Most Frequently Used with Electrolytes B, C, and D (see Table I) (× 0.25). $C$ = Metallic mass of 18-8 stainless steel; $M$ = insulating sleeve; $P$ = specimen to be polished; $b$ = supporting ring of insulating envelope; $e$ = envelope of synthetic fabric; and $t$ = conducting rod.

Using the same principle, we have attempted to work out a technique permitting rapid local polishing on metallic parts made of materials currently used (carbon steels, stainless and alloy steels, copper metals, light and ultra-light metals, nickel, cobalt, refractory alloys with a base of nickel or of cobalt, chromium, uranium, zirconium, etc.). The smallest area that can be locally polished is of the order of 1 sq cm; in many cases considerably larger polished areas may be obtained.

### *Equipment:*

The equipment consists of a "plug" formed by a massive metallic electrode surrounded by some substance electrically non-conducting, impervious to electrolytic attack, and sufficiently spongy to retain an appreciable amount of the electrolyte.

Several types of metallic electrode and insulating materials have been found satisfactory. The plug shown in Fig. 1 is suitable for polishing easily accessible areas of a specimen. It is made of copper (for polishing copper metals) or of 18-8 stainless steel (for polishing all materials except those with a copper base). The insulating cover is made of sheets of either fiber glass or synthetic textile fibers (polyvinyl acetate or polyester such as Dacron). Heavy glass fiber is indispensable for polishing copper and its alloys in a phosphoric medium, but it must be covered on the outside by a synthetic fabric with a very closely woven weft. For all other materials the electrode is surrounded by two sheets of Dacron fibers, the first much heavier than the second; this substance is resistant to the elevation of temperature encountered during the course of electrolysis.

### *Electrolytes:*

Not all electrolytes available for polishing in an electrolytic cell are suitable for this particular use. Table I lists several solutions especially recommended for the type of plug described here.

*Technique of Operation:*

The source of direct current consists of a rectifier supplied by an adjustable autotransformer. The voltage must be kept between 5 and 40 v. The positive and negative poles are attached to the specimen and to the stem of the plug respectively. The terminal voltage is regulated to the most suitable strength, depending on the nature of the material, the composition of the electrolyte and, to a certain extent, on the type of plug. Proper voltage strengths are given in Table II.

TABLE I.—COMPOSITION OF ELECTROLYTES USED IN POLISHING WITH A PLUG

| Electrolyte | Composition of Base | Active Acid | Concentration, g per 100 ml of solution |
|---|---|---|---|
| A | Orthophosphoric acid - ethanol | $H_3PO_4$ | 34 |
| B | Nitric acid - methanol | $HNO_3$ | 28 |
| C-1 | Perchloric acid - acetic acid | $HClO_4$ | 5.7 |
| C-2 | | $HClO_4$ | 7.6 |
| C-3 | | $HClO_4$ | 9 to 11 |
| C-4 | | $HClO_4$ | 23.7 |
| C-5 | | $HClO_4$ | 26.6 |
| D | Perchloric acid- methanol | $HClO_4$ | 16 |

TABLE II.—ELECTRICAL REQUIREMENTS FOR POLISHING WITH A PLUG.

| Metal | Electrolyte | Electric Potential, v |
|---|---|---|
| Copper and copper alloys | A | 10 to 15 |
| | B | 15 to 20 |
| Iron, carbon steels, austenitic manganese steels | C-3 | 30 to 40 |
| Nickel-chromium ferritic and austenitic stainless steels | C-4 | 25 to 35 |
| Aluminum and light alloys | D | 20 to 30 |
| Chromium | C-1 and D | 30 |
| Magnesium and its alloys | D | 15 to 20 |
| Nickel, cobalt, and their alloys | C-3 | 25 to 30 |
| 80-20 Nickel-chromium alloys with additions of titanium, aluminum, and cobalt (Nimonic types 75, 80, 90, 95, 100) | C-3 | 25 to 30 |
| Uranium, zirconium | D and C-3 | 20 to 25 |

The covering of the plug is saturated with the electrolyte by simple immersion. In the course of electrolysis the electrolyte is replenished in the same manner, but in general the amount of liquid must be kept small enough that it does not spread too far beyond the point of contact. The intensity of the current depends on the factors listed above and also on the amount of electrolyte. The current is strongest at the time of initial contact and tends to weaken from then on. It is usually held between 0.2 and 0.6 amp.

Copper and its alloys are the materials most easy to polish with the plug technique because the high conductivity of the phosphoric electrolyte prevents the local rise in temperature produced with solutions containing perchloric acid, used for all other materials. By slow movement of the plug an excellent polish can be extended over as large an area as desired. If the polishing is to be restricted to the point of contact of the plug, it is accomplished by successive rapid and light contacts of the plug to avoid the appearance of thread marks from the fabric. This same type of defect is apt to occur with all materials and with all electrolytes.

The nitric acid - methanol electrolyte gives equally good results with copper and copper alloys, using a plug of stainless steel. It has the advantage of reducing perceptibly the length of the operation. Furthermore, this electrolyte is the only one that is suitable for brasses containing iron, and for copper-nickel alloys.

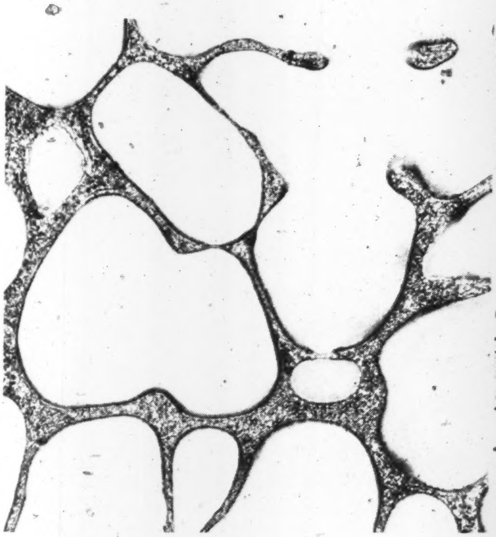

FIG. 2.—Shell Casting of 8 per cent Copper Aluminum Alloy (× 160). Polished 45 sec with electrolyte D at 20 v.

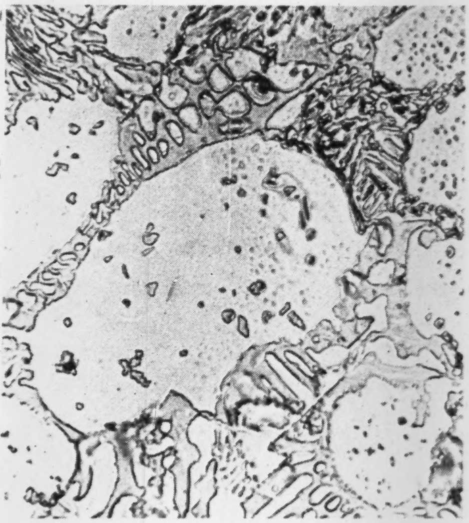

FIG. 3.—Cast Oneral Alloy (Cobalt, 50 per cent; Chromium, 26 per cent; Molybdenum, 10 per cent; Nickel, 6 per cent; Iron, 4 per cent) (× 400). Polished 30 sec with electrolyte C-3 at 27 v.

The polishing of aluminum and its light alloys requires special precaution. The terminal voltage is rather critical, since below a certain point the effect of the polishing is replaced by anodic oxidation and by etching, causing white spots. Even when the voltage is correct these phenomena tend to occur on the first contact of the plug if it is held in a fixed position. One can avoid them by rapid movement over a distance of about 2 to 3 cm. When the area is thus made perfectly smooth the final polish can be produced by successive slow applications to the particular area selected. Sometimes this technique, which works well with pure and commercial aluminums, tends to induce etching effects on mixed alloys. In this case one would from the beginning use successive quick touches of the plug for several seconds, then slower applications.

Polishing time depends on the initial condition of the surface and the size of the area to be polished. When polishing is limited only to the point of contact of the plug, say about 1 sq cm, and with a prior abrasion to No. 00 emery paper, this time is of the order of 30 to 60 sec, less than that required for electrolytic cell polishing. This speed, which compares favorably with the ultra-rapid polishing on commercial apparatus (6) is due to the density of the relatively high anodic current. Larger areas obviously require a more prolonged electrolysis since the plug must be moved about.

When the local electrolysis is ended, the surface is rinsed thoroughly with water, then with alcohol, then dried in a jet of warm air. If the shape and size of the specimen do not permit rinsing under running water, a simple jet will suffice and, if necessary, one can omit this step and be content with a rapid irrigation with alcohol. When the material is hard enough that there is no danger of scratching, drying can be done with a sheet of filter paper.

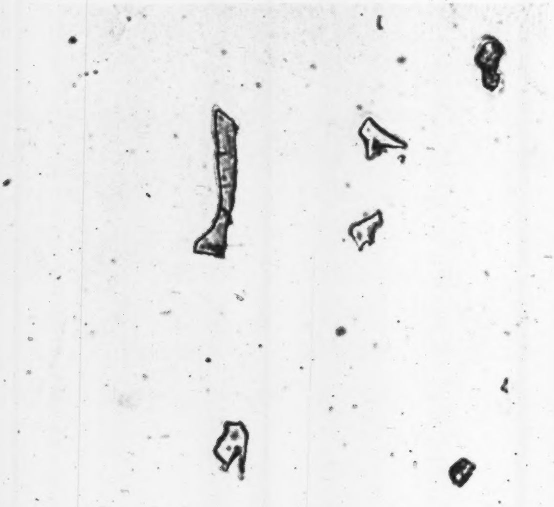

FIG. 4.—Longitudinal Section of a Forged Bar Showing Inclusions in a Nimonic-100 Alloy (80-20 Nickel-Chromium Alloy with Additions of Titanium, Aluminum, and Cobalt) (× 1000). Polished 30 sec with electrolyte C-3 at 30 v.

Periodically, the metallic electrode is cleaned by washing and light rubbing. The fabric envelopes remain in good condition if one takes the precaution of rinsing them carefully in water after a certain amount of continuous service. However, the polishing of copper with an alcoholic solution of phosphoric acid is an exception because the metal dissolved from the anode is deposited on the cathode in the form of a powdery layer which finally permeates the insulating envelope and causes a short circuit, preventing a correct polishing. This occurs much more quickly when the envelope is somewhat thin; for this reason the metallic mass is surrounded by a layer of very heavy glass which is in turn covered with a closely woven

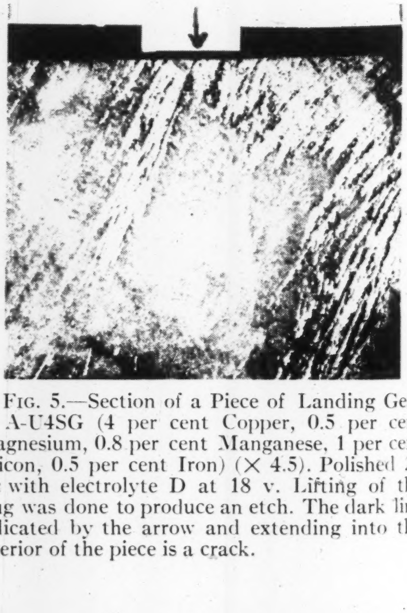

FIG. 5.—Section of a Piece of Landing Gear of A-U4SG (4 per cent Copper, 0.5 per cent Magnesium, 0.8 per cent Manganese, 1 per cent Silicon, 0.5 per cent Iron) (× 4.5). Polished 20 sec with electrolyte D at 18 v. Lifting of the plug was done to produce an etch. The dark line indicated by the arrow and extending into the interior of the piece is a crack.

synthetic fabric intended to separate the fragile glass fibers from the surface to be polished. With such an arrangement the deposit of copper saturates the fabrics at the end of about 15 min of electrolysis, the time sufficient to polish a considerable area. Then the fabrics must be replaced because the deposit is heavily encrusted on the threads and resists all attack. It must be noted that the re-deposit of copper is not produced with the nitric acid - methanol electrolyte.

*Results:*

Although devised for the preparation of surfaces intended for the taking of replicas, plug polishing is applicable, of course, to ordinary metallic specimens. It has been shown to be especially practical for the examination of flat or slightly curved sections too massive to be treated easily in an electrolytic cell but nevertheless small enough to be placed on the stage of a microscope. In addition to its speed, the method has the great advantage of limiting the polishing only to areas of interest.

The quality of the polish obtained is good and structural differences show up in the finest detail and without any relief effects (Figs. 2, 3, and 4). In the case of copper metals, grain boundaries can be brought out by lowering the terminal voltage at the end of the electrolysis. In the same way, and by moving the plug, one produces an effect of macro-etching on certain light alloys (Fig. 5). However, in general the microstructures will be brought into evidence with the usual chemical reagents, as shown in Fig. 6, which shows the transition zone in a section of bonded steel. (On a specimen measuring 90 by 50 by 15 mm, the area selected had been heavily abraded, polished with the plug for 2 min, then etched 10 sec with Benedick's reagent (Metani-

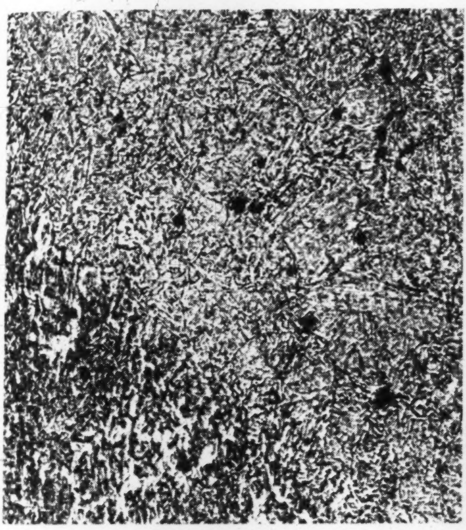

FIG. 6.—Local Polishing for 30 sec on an Assembly of Two Welded Sheets of Steel Measuring 100 by 50 by 10 mm; Electrolyte C-3 Was Used at 35 v (× 160). Etched 5 sec with Benedick's reagent. The micrograph shows the line between the weld (at the left) and the transition zone (at the right).

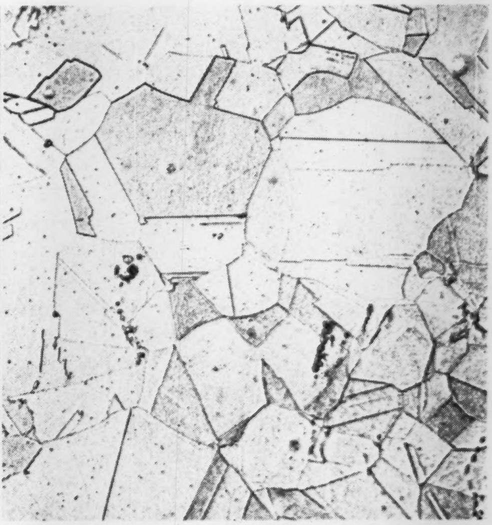

FIG. 7.—Surface of a Sheet of 18-8 Stainless Steel 1 mm Thick (× 400). Polished 60 sec with electrolyte C-4 at 25 v; etched 5 sec with Marble's reagent.

trobenzine sulfonic acid in alcohol.) Figure 7 shows another example of plug polishing and etching (with Marble's reagent containing 4 g $CuSO_4$, 20 ml concentrated HCl, and 20 ml $H_2O$) of a sheet of annealed 18-8 stainless steel measuring 200 by 80 mm, much too large to be polished in an electrolytic cell.

In conclusion, local electrolytic polishing with a plug is superior in nearly every instance to laboratory electrolytic cell facilities used up to the present for the preparation of metallographic specimens. It is obviously also applicable to the preparation of specimens intended for studies of fatigue, corrosion, friction, etc. Its greatest advantages are (1) the speed of operation and (2) the possibility of treating only selected spots on objects of any shape or dimensions. The technique has been successfully used in practical applications with steels and with aluminum, cobalt, chromium, copper, nickel, uranium, and zirconium and their alloys.

## REPLICA TECHNIQUE

### *General Considerations:*

Development of the replica method to be described had its origin in an attempt to discover and follow, over a period of time, fatigue cracks on pieces of aircraft. A. Van Effenterre, commandant of a naval air base in France, thought of using a transparent replica method of the type well known in electron micrography, but applying it to surfaces of very large dimensions. Systematic trials enabled him to select a cellulosic lacquer, then to determine the best means of application, removal of the film, and examination. The remarkable results obtained in the solution of that particular problem prompted us to extend the technique to all areas of macro- and micrography on large pieces and on metallographic specimens.

### *Type of Lacquer:*

Numerous attempts showed that lacquer of the collodion type did not give results sufficiently reproducible because of the formation of gas bubbles caused by the low boiling point of the solvent. On the other hand, the Faxfilm method (1), already known, could not be applied to surfaces of large dimensions or to surfaces presenting extreme curvature. The most satisfactory material is a cellulosic compound dissolved in a mixture of solvents and appropriate plastifier. This lacquer, called "Replic," lasts for at least 6 months without losing its properties.

### *Application of Lacquer:*

The lacquer is used in a form more or less rich in solvents according to the size of the surface to be covered. Surfaces of large dimensions require a liquid of greater viscosity, otherwise the solid film is apt to be too thin.

All methods of application—immersion, direct spreading, painting with a brush, spreading with a spatula—are possible. However, the most recommended method consists of making the liquid flow on a slightly inclined surface. The excess will settle in a heavier mass toward the opposite end; therefore the spreading must be done away from the area which will eventually show in the replica. When the shape or dimension of the piece does not lend itself to this mode of application, it suffices to pour on a sufficient amount of lacquer and to spread it with the finger or with a spatula, taking care not to touch the metal. Spreading with a brush risks the introduction of very fine air bubbles, but this is no disadvantage in replicas intended only for macroscopic examination.

On larger surfaces, the film may be too thin after drying. In this case, another coat of lacquer is applied.

In order to limit the amount of lacquer and to facilitate the ultimate removal of the film, it is practical to surround the area to be reproduced with an adhesive tape (Scotch tape), of which half the width is pasted on the surface and the other half is held vertically.

*Removal of the Film:*

After application of the lacquer, the specimen is exposed to the air until completely dry. The time necessary depends on the atmospheric temperature; at about 20 C, at least 45 to 60 min is needed. Quick drying with an oven or under infra-red rays must be used with caution because of risk of deterioration of the film if the temperature goes above 35 C.

Removal of the film is begun at the thickest end; the film is lifted with a razor blade or a biologist's lance-shaped needle, then pulled with forceps with an even, steady movement. To avoid tears it is important that traction be exerted evenly on the entire length of the edge selected. The adhesive tape mentioned above works admirably in this connection.

The film is sometimes especially adherent on certain surface conditions. When sufficiently thick to resist traction without breaking, the film can no longer be detached by a regular movement, and defects or artifacts result, consisting of parallel striations very annoying in micrographic examination. There is a very simple means of avoiding both the beginning of rupture and the artifacts of mechanical origin. From the moment traction shows too great adherence, freeing of the film is limited to a band several millimeters in width, then the entire surface of the film is softened with water with a very small amount of Teepol[3] added. This softening is done either by immersion or, if the piece is large, by application with a cotton sponge. After a certain length of contact which may vary from several minutes to an hour or more, light traction will effect the complete removal of the film. This maneuver is indispensable in the following three cases: (1) when the area covered with lacquer contains deep cracks likely to initiate tearing; (2) when the firm adherence results in an irregular detachment, producing striations; (3) when the film is too thin to resist any mechanical effort.

Softening with diluted Teepol has two disadvantages. For one, there is danger of spotting the metallic surface, which is troublesome when it must receive other replicas without intermediate polishing (for example, in the case of fatigue specimens). In the second place, the softened film must be rinsed with distilled water and dried. To reduce the first disadvantage as much as possible, length of contact with the liquid must be held to a minimum, and the specimen must be rinsed and dried immediately after detachment. Rinsing and drying of the film require certain precautions to avoid impurities or dust settling on the side bearing the imprint of the surface. For these reasons we are currently studying another procedure consisting of spreading a thin oily film over the surface before the taking of a replica.

*Mounting for Examination:*

The dry film tends to curl (the concave side is the one bearing the imprint), so it cannot be studied without preliminary mounting. There are several means of accomplishing such mounting, and one should select the one which is the most simple and the best adapted to the particular case.

1. The replica is placed between two sheets of cellophane, or better, in an envelope of thin cellulose acetate. This

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[3] A higher alkyd sulfate used as a wetting agent and detergent.

method of keeping the films free from dust permits examination by transmitted or reflected light under low and medium magnifications. In particular, it is well suited to projection on a screen with the help of a lamp, a film projector, a profilometer, etc.

2. The replica is placed on a sheet of clean glass, with the face to be examined turned toward the top (this face is easily recognized since it is decidedly less shiny than the opposing face), and it is held in place by placing adhesive tape on its four sides, or on two opposite sides. This method is applicable only to replicas taken on flat surfaces or easily reproducible (cylindrical) surfaces.

3. Slippery replicas can be attached to the glass by simple softening with distilled water and pressure with a roller made of a glass rod encased in a tube of perfectly clean rubber. This method, which is practicable for large replicas, does not insure a perfect mounting.

4. Various types of adhesives have been tried with varying results. It is obviously essential that the substances of which the adhesives are made have no detrimental action on the film. Adhesives with a gum arabic or coumarone base are useful, but the one which seems the best, and in most general use when the replica is taken on a very wrinkled surface or on a shape not perfectly reproducible, is of the glycerinated gelatin well known to microbiologists. The recommended technique is as follows.

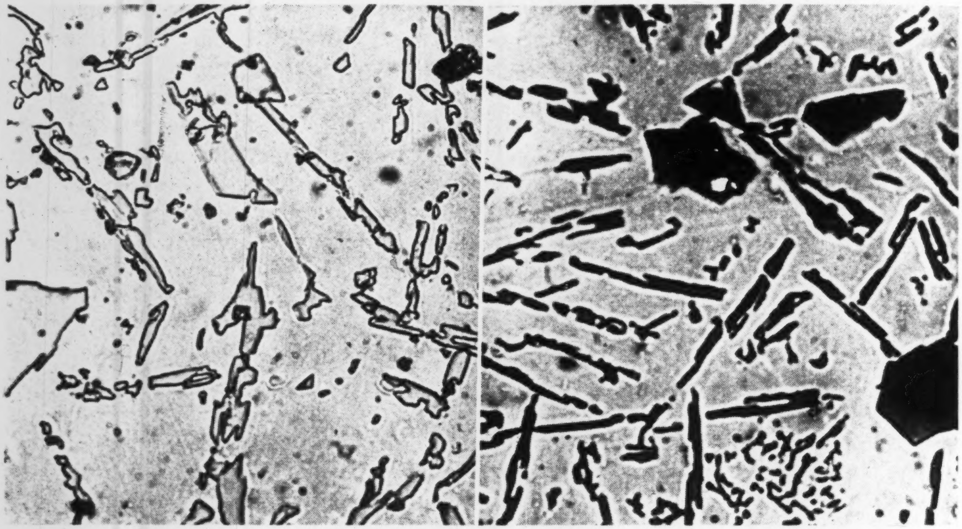
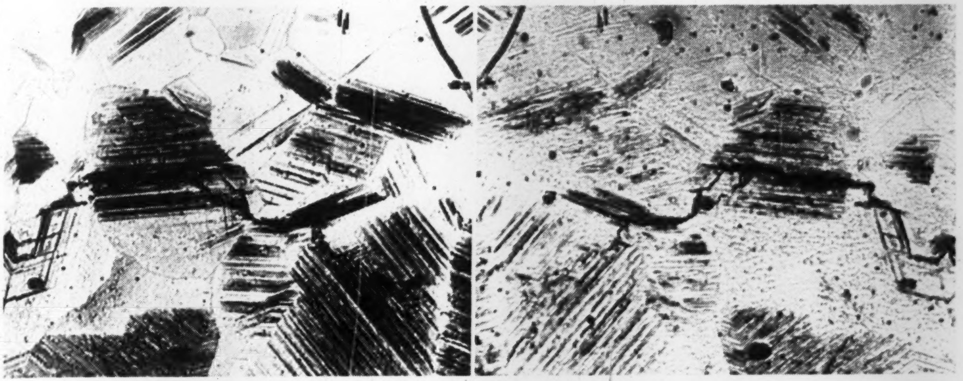

(*a*) Untouched replica. (*b*) replica stained with nigrosin.

FIG. 8.—Replica of an Aluminum Alloy Polished Electrolytically with the Disa-Electropol (a Commercial Electrolytic Polishing Apparatus) (× 400). The photograph was taken by transmitted light on an ordinary microscope

The glycerinated gelatin is brought to liquid consistency by immersing the flask in water at 70 to 80 C (a temperature above 80 C leads to the appearance of bubbles which would make the substance useless). With the help of a rod, the liquid is spread over the glass plate in sufficient quantity to cover the entire surface of the film. Its under face is covered immediately, and then extended to reach each of its edges. A rubber-covered glass rod is rolled over the free surface; this drives out any air bubbles and pushes toward the edges the excess of still semi-liquid glue that is subsequently removed by abrasion after complete solidification. The preparation is placed under running water and rubbed with a soft brush or a cotton sponge. Thorough cleansing is indispensable, and it is well to control this by checking that the microscopic appearance after drying is the same after a second washing. If not, it is because of particles of solid adhesive still adhering to the film. Drying is done either by a current of filtered, unheated air or with filter paper free of lint or threads.


(*a*) Surface of the specimen

(*b*) same field on a replica showing a crack indiscernible in (*a*).

FIG. 9.—Specimen of Alpha Brass Polished Electrolytically, Then Submitted to Fatigue Tests (Alternating Stress) (× 160).

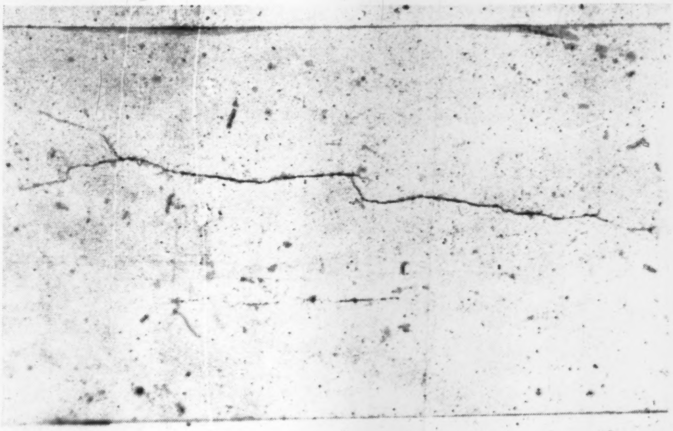


FIG. 10.—Replica Taken on a Cylindrical Specimen of Steel 58 mm in Diameter Submitted to Fatigue Tests by Alternating Torsion (Natural size). Direct photography on a profilometer.

*Details of Examination:*

The films are easily charged with static electricity, with a strong attraction for dust as a result. They must be handled and stored with certain precautions as to cleanliness. Wiping with Electrowipe[4] tissue constitutes an effective dry cleaning.

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[4] An antistatic agent.

During examination with an ordinary microscope, a simple technique well known to microbiologists allows one to increase greatly the contrasts in the outline details of the replica. It consists of spreading over the preparation a suspension of special India ink or a solution of nigrosin; the black color accumulates in the depressed areas which then appear dark against a light background (Fig. 8).

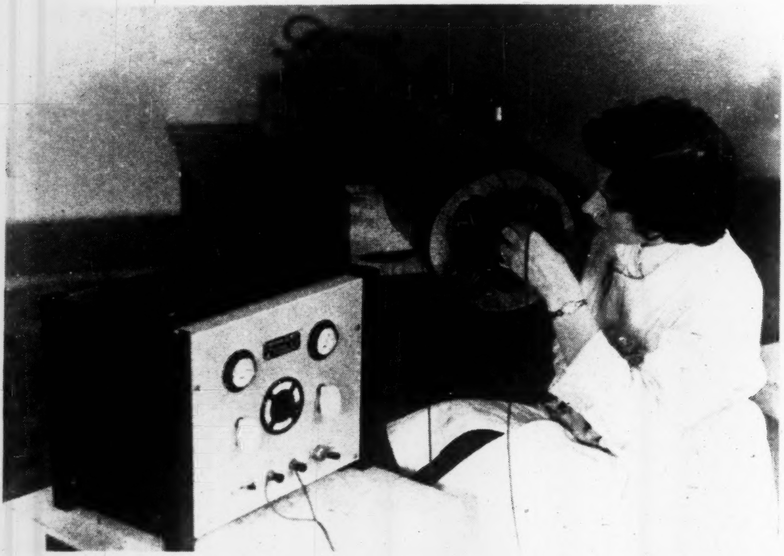

FIG. 11.—Plug Polishing of the Interior Diameter of a Broken Axle.

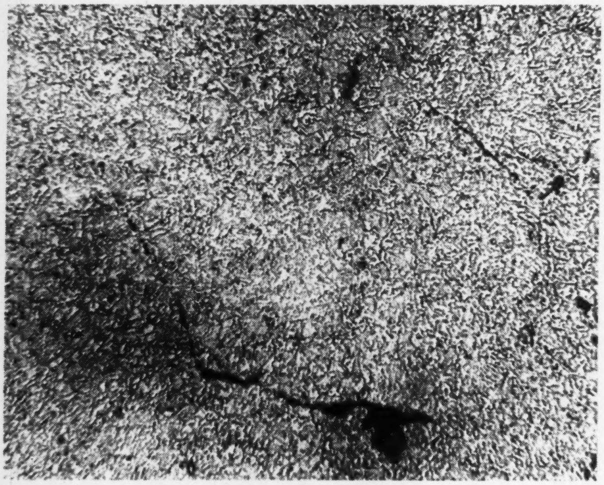

FIG. 12.—Micrograph of the Replica Taken on the Interior Diameter of the Axle Shown in Fig. 11, After Plug Polishing and Etching (× 63). Typical structure of overheated steel.

The surface of transparent replicas can be examined by reflected light, exactly as is done with an opaque body. The only difference is that the luminosity is not as good as that given by the corresponding metallic specimen. This loss of luminosity results in a certain increase of exposure time needed for photography. Two means of remedying this defect are described below:

1. A highly polished surface, a silvered mirror, for example, is placed behind the replica, that is, on the glass slide which supports it. This greatly increases the amount of reflected light, and also the contrast in certain details of micro-outline. On the other hand it can diminish the contrast of other details. Thus, under magnifications of 100 to 400 diameters, very fine cracks are much clearer with the mirror, while the microconstituents of an alloy often show up less well. Therefore, as a general rule one will select the means of study best suited to the end in view.

For macrography by reflected light the increase in luminosity is always accompanied by better definition of the contrasts in half-light. To obtain the maximum effect, the mirror must not be placed against the glass, but slightly higher and not strictly parallel to the stage. The two variable factors of distance and direction are easily regulated by resting the four corners of the mirror on soft wax.

2. Metalization of the anterior face of the replica, by the technique of vaporization under vacuum used in electron micrography, presents considerable advantages: (1) The plastic is isolated from the atmosphere, facilitating its preservation; (2) the reflecting power is considerably superior to that of the original metallic surface; (3) the replica can be examined under higher magnifications with oil-immersion objectives.

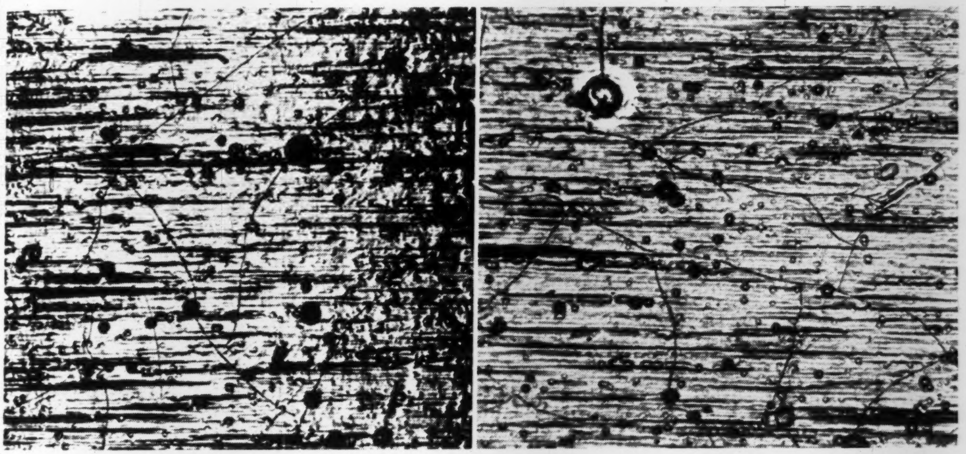
(*a*) (*b*)

FIG. 13.—Comparison of the Surface (*a*) and the Replica (*b*) of an Axle of Hardened Steel Coated with 50 $\mu$ of Hard Chromium, Then Polished Electrolytically with the Plug (× 63). Because of the small diameter of the axle (15 mm) the direct micrograph is defective. In contrast, the entire surface of the piece can be photographed on the replica. The network of cracks revealed by polishing the chrome can be seen.

The optical techniques of phase contrast and interference contrast with two polarized waves (7) can be used with replicas as with opaque surfaces, and have the same sensitivity of resolution of details of micro- and submicro-outlines. For such examinations, and especially under very high magnifications, metalization with aluminum is of great interest. Finally we call attention to a very curious phenomenon observed with replicas taken on certain types of surface, then metalized: When the metallic specimen is active when opposite the polarized light, the replica is evidently inactive but acquires activity after deposition of the aluminum film.

## CONCLUSION

The study of the applications of the Replic lacquer has only a short history (a few months), and yet the practical results already obtained are numerous and varied. It has been found that the replica provides a faithful reproduction of surfaces in their actual state as well as polished metallographic structures (Figs. 9 to 13). The technique has been successfully applied to the study of diffusion zones in alloys, broken structural parts, propagation of fatigue cracks, thermal shock cracks in turbine blades, corrosions, nuclear fuel elements, and numerous other solids.

The procedures of local electrolytic polishing and replication are deserving of close attention by metallographers and persons interested in nondestructive testing.

## REFERENCES

(1) Described in R. D. Johnson, J. R. Morgan, and G. R. Mallet, NACA Technical Note 3351, April 1955.

(2) M. S. Hunter and W. G. Fricke, Jr., "Metallographic Aspects of Fatigue Behavior of Aluminum," Am. Soc. Testing Mats., Vol. 54, p. 717 (1954).

(3) T. K. Bierlein, J. R. Morgan, and G. R. Mallet, "The Metallography of Irradiated Uranium—Preparation, Cathodic Vacuum Etching, and Replication," Atomic Energy Commission Report HW-42184 Rev., May 25, 1956.

(4) P. A. Jacquet, *Nature*, Vol. 135, p. 1076 (1935); *Bulletin*, Société Chimique de France, Series 5, Vol. 3, p. 705 (1936); *Revue de Métallurgie*, Vol. 37, pp. 210, 244 (1940).

(5) J. Mazia, *Steel*, Vol. 120, p. 84, May 19, 1947.

(6) P. A. Jacquet, "Métallographie de l'aluminium et de ses alliages. Emploi du polissage électrolytique," O.N.E.R.A. Publication No. 51, April, 1952; "Metallography of Aluminum and Its Alloys. Use of Electrolytic Polishing," N.A.C.A. Technical Memorandum 1384, Nov., 1955.

(7) G. Nomarski and A. R. Weill, *Revue de Métallurgie* Vol. 52, p. 121 (1955).

## DISCUSSION

MR. WILLIAM G. FRICKE, JR[1].—For some time we at the Alcoa Research Laboratories have been using replica methods for the reasons outlined by Mr. Jacquet.

One great advantage of replication is that you can go back and look at the replicas after you know where to look. We are obtaining them, for example, from fatigue specimens during a test. After cracks appear, we can examine replicas made previously to observe the early stages of fatigue in these areas.

In our latest investigation,[2] in order to examine cracks in notched fatigue specimens it was necessary to resort to replication because the working distance of high-powered microscope lenses was not enough to bring the base of the notch into focus.

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[1] Research Metallurgist, Alcoa Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] M. S. Hunter and W. G. Fricke, Jr., "Cracking of Notch Fatigue Specimens," p. 643, this publication.

# THE SIGNIFICANCE OF TEST RESULTS FROM SMALL GROUPS OF SPECIMENS*

By Evan H. Schuette[1]

## Synopsis

A method is presented for defining the significance, in terms of both confidence levels and expected "exceedances," of the lowest results obtained from small sample groups of equal size. The method is then extended to apply as well to the second and higher ranking results. Development is independent of the distribution form of the population sampled.

Methods are presented for comparing groups of unequal size on the basis of equal significance. A table gives plotting positions on probability paper, permitting interpolation of expected "exceedance" at any given confidence level.

In an earlier paper,[2] it was shown that if fatigue tests—or life tests in general—are conducted in groups of $n$ specimens each, with only the lowest of the $n$ test results being recorded, then the median of such "least-of-$n$" results will be a value that may be expected to be exceeded by a percentage of the parent population given by $\frac{1}{2}^{(1/n)} \times 100$.

Because this procedure, besides reducing the scatter that must be dealt with, has the effect of increasing the useful significance of the data without resort to statistical analysis, it has stimulated a considerable interest among industrial researchers in particular. However, the original work left some important questions unanswered, for example: what is the confidence level associated with the predictions made? How much spread is to be expected between lines or points for two different values of "exceedance"? How would the line or point be altered by the imposition of other confidence levels?

Further refinements in handling the applicable probability functions have revealed that answers to these and other questions can be provided without unduly complicating the job of the test engineer. For the case of a single result with all test conditions fixed, the analysis possesses complete mathematical rigor. Extension to the determination of a life curve (for example, the $S$-$N$ curve in fatigue) requires some departure from complete rigor, but it is possible to make the extension on an intuitive basis with reasonable accuracy.

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[2] Evan H. Schuette, "A Simplified Statistical Procedure for Obtaining Design-Level Fatigue Curves," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 853 (1954).

In the present paper, the extensions to the first analysis are outlined, and tables are presented that are suitable for use in a variety of data-handling problems.

## PROBABILITY ANALYSIS WITH CONSTANT TEST CONDITIONS

Given some undefined frequency distribution (Fig. 1) for the possible results of a test in terms of the independent variable, $x$, it is implicit by definition that the probability for occurrence of a value between $x_1$ and $x_2$ is given by the area included under the frequency-distribution curve between these two values. The total area under the curve is unity, since a result somewhere between its two end limits (whether infinite or finite) is a certainty, or has a probability of one. Likewise, the probability of a result greater than any particular value of $x$ is given by $A_x$, the area included under the curve to the right of $x$.

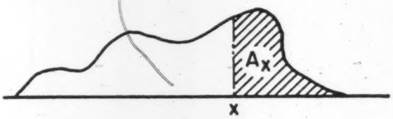

FIG. 1.—Hypothetical Frequency Distribution for the Variable $x$.

If $n$ tests are conducted, the probability in each test of exceeding the value $x$ is $A_x$; the probability that all $n$ tests will give values greater than $x$ is, by elementary probability theory, $A_x^n$.

Let us now ask what is the chance that a proportion less than $B$ (an arbitrary figure) of all possible future test results will fall above the "least-of-$n$" value obtained from a series of $n$ tests. This is precisely the same as the probability that all $n$ test results would fall above the value $x$ that defines an area $A_x = B$, or

$$p = A_x^n$$

A simple numerical example will serve to illustrate the significance of the above equation. If we have determined the lowest value from five identical tests, and now wish to know the chance that less than half of the population lies above this point, we calculate

$$p = 0.50^5$$
$$= 0.031$$

Thus, there is only 3.1 per cent risk in saying that 50 per cent or more of the population exceeds our "least-of-five" value. Conversely, the confidence level for such a statement is 96.9 per cent.

Herein lies the major extension, mathematically, of the present paper over the previous one.[2] In the earlier work the possibility of calculating the confidence level associated with any given "exceedance" prediction was overlooked. The "exceedance" corresponding to a median "least-of-$n$" result was calculated without recognizing that this also represented a 50 per cent confidence level, and that other values could be obtained from the same simple equation.

### *Analysis of Higher Ranking Points:*

If other than the lowest value from a given group of tests have been determined, similar statements can be made regarding the second and higher ranking points. The chance that at least four out of five values lie above a given value, $x$, is the chance that all five lie above, plus the individual chances for any one to fall below, while the other four lie above. This total probability is given by

$$p = A_x^5 + 5A_x^4(1 - A_x)$$

$A_x$ has the same significance as in the previous analysis and in Fig. 1. In the general case, the equation becomes

$$p = A_x^n + nA_x^{(n-1)}(1 - A_x)$$

Continuing with the previous numeri-

### TABLE I.—CONFIDENCE LEVELS AND "EXCEEDANCES" FOR GROUPED SAMPLES

| Number of Points per Group, *n* | Confidence Level, per cent | Minimum Per Cent of Population Exceeding Median of: | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | LOWEST RANKING POINTS | | | | | | | SECOND RANKING POINTS | | | | | | |
| | | 1 group | 3 groups | 5 groups | 7 groups | 9 groups | 15 groups | 25 groups | 1 group | 3 groups | 5 groups | 7 groups | 9 groups | 15 groups | 25 groups |
| 1... | 50 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | ... | ... | ... | ... | ... | ... | ... |
| | 75 | 25.00 | 32.64 | 35.94 | 37.88 | 39.20 | 41.50 | 43.35 | ... | ... | ... | ... | ... | ... | ... |
| | 90 | 10.00 | 19.58 | 24.66 | 27.86 | 30.10 | 34.15 | 37.51 | ... | ... | ... | ... | ... | ... | ... |
| | 95 | 5.00 | 13.53 | 18.94 | 22.53 | 25.14 | 30.00 | 34.14 | ... | ... | ... | ... | ... | ... | ... |
| | 98 | 2.00 | 8.40 | 13.52 | 17.27 | 20.10 | 25.61 | 30.48 | ... | ... | ... | ... | ... | ... | ... |
| | 99 | 1.00 | 5.89 | 10.56 | 14.23 | 17.10 | 23.14 | 28.14 | ... | ... | ... | ... | ... | ... | ... |
| | 99.9 | 0.10 | 1.84 | 4.76 | 7.67 | 10.25 | 16.12 | 22.06 | ... | ... | ... | ... | ... | ... | ... |
| 3... | 50 | 79.37 | 79.37 | 79.37 | 79.37 | 79.37 | 79.37 | 79.37 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 |
| | 75 | 63.00 | 68.84 | 71.10 | 72.35 | 73.18 | 74.58 | 75.68 | 32.64 | 38.20 | 40.51 | 41.84 | 42.74 | 44.30 | 45.55 |
| | 90 | 46.42 | 58.06 | 62.71 | 65.31 | 67.01 | 69.89 | 72.12 | 19.58 | 28.37 | 32.38 | 34.76 | 36.39 | 39.26 | 41.59 |
| | 95 | 36.84 | 51.34 | 57.41 | 60.85 | 63.11 | 66.94 | 69.89 | 13.53 | 23.09 | 27.82 | 30.73 | 32.73 | 36.32 | 39.26 |
| | 98 | 27.14 | 43.80 | 51.33 | 55.68 | 58.57 | 63.50 | 67.29 | 8.40 | 17.83 | 23.08 | 26.43 | 28.79 | 33.09 | 36.67 |
| | 99 | 21.54 | 38.90 | 47.27 | 52.20 | 55.50 | 61.15 | 65.53 | 5.89 | 14.75 | 20.16 | 23.73 | 26.28 | 31.00 | 34.97 |
| | 99.9 | 10.00 | 26.38 | 36.22 | 42.47 | 46.80 | 54.41 | 60.42 | 1.84 | 8.04 | 13.18 | 16.97 | 19.84 | 25.43 | 30.36 |
| 4... | 50 | 84.09 | 84.09 | 84.09 | 84.09 | 84.09 | 84.09 | 84.09 | 61.42 | 61.42 | 61.42 | 61.42 | 61.42 | 61.42 | 61.42 |
| | 75 | 70.71 | 75.58 | 77.42 | 78.45 | 79.12 | 80.26 | 81.14 | 45.63 | 50.91 | 53.04 | 54.25 | 55.06 | 56.45 | 57.55 |
| | 90 | 56.23 | 66.52 | 70.47 | 72.65 | 74.06 | 76.44 | 78.26 | 32.05 | 41.40 | 45.38 | 47.68 | 49.22 | 51.90 | 54.02 |
| | 95 | 47.29 | 60.65 | 65.95 | 68.89 | 70.80 | 74.00 | 76.43 | 24.86 | 35.91 | 40.85 | 43.77 | 45.73 | 49.16 | 51.88 |
| | 98 | 37.61 | 53.84 | 60.64 | 64.46 | 66.95 | 71.13 | 74.30 | 17.94 | 30.04 | 35.90 | 39.43 | 41.83 | 46.07 | 49.48 |
| | 99 | 31.62 | 49.26 | 57.01 | 61.41 | 64.30 | 69.15 | 72.83 | 14.09 | 26.38 | 32.71 | 36.59 | 39.27 | 44.03 | 47.87 |
| | 99.9 | 17.78 | 36.81 | 46.69 | 52.61 | 56.58 | 63.36 | 68.53 | 6.40 | 17.41 | 24.41 | 29.04 | 32.34 | 38.38 | 43.40 |
| 5... | 50 | 87.05 | 87.05 | 87.05 | 87.05 | 87.05 | 87.05 | 87.05 | 68.61 | 68.61 | 68.61 | 68.61 | 68.61 | 68.61 | 68.61 |
| | 75 | 75.79 | 79.93 | 81.49 | 82.35 | 82.91 | 83.87 | 84.60 | 54.58 | 59.38 | 61.28 | 62.35 | 63.07 | 64.29 | 65.26 |
| | 90 | 63.10 | 72.17 | 75.58 | 77.44 | 78.65 | 80.66 | 82.19 | 41.61 | 50.65 | 54.35 | 56.46 | 57.85 | 60.26 | 62.15 |
| | 95 | 54.93 | 67.03 | 71.68 | 74.22 | 75.86 | 78.59 | 80.65 | 34.26 | 45.40 | 50.13 | 52.85 | 54.67 | 57.79 | 60.25 |
| | 98 | 45.74 | 60.93 | 67.02 | 70.38 | 72.55 | 76.15 | 78.85 | 26.71 | 39.60 | 45.40 | 48.78 | 51.05 | 54.98 | 58.09 |
| | 99 | 39.81 | 56.75 | 63.79 | 67.70 | 70.23 | 74.44 | 77.60 | 22.21 | 35.84 | 42 26 | 46.07 | 48.64 | 53.10 | 56.63 |
| | 99.9 | 25.12 | 44.95 | 54.37 | 59.82 | 63.41 | 69.41 | 73.91 | 12.20 | 26.10 | 33.78 | 38.58 | 41.90 | 47.79 | 52.52 |
| 6... | 50 | 89.09 | 89.09 | 89.09 | 89.09 | 89.09 | 89.09 | 89.09 | 73.55 | 73.55 | 73.55 | 73.55 | 73.55 | 73.55 | 73.55 |
| | 75 | 79.37 | 82.97 | 84.32 | 85.06 | 85.54 | 86.36 | 86.99 | 61.05 | 65.38 | 65.08 | 68.03 | 68.67 | 69.76 | 70.61 |
| | 90 | 68.13 | 76.20 | 79.19 | 80.81 | 81.86 | 83.60 | 84.92 | 48.97 | 57.45 | 60.84 | 62.75 | 64.01 | 66.17 | 67.86 |
| | 95 | 60.70 | 71.65 | 75.77 | 78.00 | 79.44 | 81.81 | 83.60 | 41.82 | 52.56 | 56.97 | 59.48 | 61.13 | 63.96 | 66.16 |
| | 98 | 52.10 | 66.18 | 71.64 | 74.62 | 76.53 | 79.68 | 82.03 | 34.17 | 47.04 | 52.56 | 55.72 | 57.82 | 61.42 | 64.23 |
| | 99 | 46.42 | 62.37 | 68.75 | 72.25 | 74.49 | 78.20 | 80.95 | 29.43 | 43.38 | 49.59 | 53.19 | 55.58 | 59.70 | 62.91 |
| | 99.9 | 31.62 | 51.36 | 60.19 | 65.17 | 68.41 | 73.76 | 77.73 | 18.14 | 33.54 | 41.35 | 46.06 | 49.25 | 54.80 | 59.17 |
| 10... | 50 | 93.30 | 93.30 | 93.30 | 93.30 | 93.30 | 93.30 | 93.30 | 83.77 | 83.77 | 83.77 | 83.77 | 83.77 | 83.77 | 83.77 |
| | 75 | 87.06 | 89.40 | 90.27 | 90.74 | 91.05 | 91.58 | 91.98 | 75.26 | 78.28 | 79.45 | 80.09 | 80.52 | 81.25 | 81.82 |
| | 90 | 79.43 | 84.95 | 86.93 | 88.00 | 88.68 | 89.81 | 90.66 | 66.31 | 72.68 | 75.11 | 76.46 | 77.34 | 78.82 | 79.97 |
| | 95 | 74.11 | 81.87 | 84.66 | 86.15 | 87.10 | 88.65 | 89.81 | 60.59 | 69.07 | 72.33 | 74.14 | 75.32 | 77.30 | 78.82 |
| | 98 | 67.62 | 78.06 | 81.86 | 83.89 | 85.17 | 87.26 | 88.79 | 53.98 | 64.81 | 69.06 | 71.42 | 72.95 | 75.52 | 77.48 |
| | 99 | 63.10 | 75.33 | 79.86 | 82.28 | 83.80 | 86.28 | 88.09 | 49.64 | 61.87 | 66.79 | 69.54 | 71.31 | 74.30 | 76.57 |
| | 99.9 | 50.12 | 67.05 | 73.74 | 77.34 | 79.63 | 83.31 | 85.97 | 37.63 | 53.40 | 60.19 | 64.02 | 66.53 | 70.73 | 73.92 |
| 20... | 50 | 96.59 | 96.59 | 96.59 | 96.59 | 96.59 | 96.59 | 96.59 | 91.75 | 91.75 | 91.75 | 91.75 | 91.75 | 91.75 | 91.75 |
| | 75 | 93.30 | 94.55 | 95.01 | 95.26 | 95.43 | 95.69 | 95.90 | 87.10 | 88.77 | 89.41 | 89.77 | 90.00 | 90.40 | 90.70 |
| | 90 | 89.12 | 92.17 | 93.24 | 93.81 | 94.17 | 94.76 | 95.21 | 81.90 | 85.63 | 87.01 | 87.76 | 88.25 | 89.07 | 89.70 |
| | 95 | 86.09 | 90.48 | 92.01 | 92.81 | 93.32 | 94.15 | 94.76 | 78.39 | 83.53 | 85.43 | 86.46 | 87.13 | 88.23 | 89.07 |
| | 98 | 82.23 | 88.35 | 90.48 | 91.59 | 92.29 | 93.41 | 94.23 | 74.12 | 80.99 | 83.53 | 84.90 | 85.78 | 87.24 | 88.33 |
| | 99 | 79.43 | 86.79 | 89.36 | 90.71 | 91.54 | 92.88 | 93.85 | 71.12 | 79.19 | 82.19 | 83.81 | 84.84 | 86.55 | 87.83 |
| | 99.9 | 70.79 | 81.88 | 85.87 | 87.94 | 89.23 | 91.27 | 92.72 | 62.24 | 73.74 | 78.14 | 80.52 | 82.03 | 84.51 | 86.33 |

TABLE I.—(*Concluded*).

| Number of Points per Group, *n* | Confidence Level, per cent | Minimum Per Cent of Population Exceeding Median of: | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | THIRD RANKING POINTS | | | | | | | FOURTH RANKING POINTS | | | | | | |
| | | 1 group | 3 groups | 5 groups | 7 groups | 9 groups | 15 groups | 25 groups | 1 group | 3 groups | 5 groups | 7 groups | 9 groups | 15 groups | 25 groups |
| 1... | 50 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| | 75 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| | 90 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| | 95 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| | 98 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| | 99 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| | 99.9 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 3... | 50 | 20.63 | 20.63 | 20.63 | 20.63 | 20.63 | 20.63 | 20.63 | ... | ... | ... | ... | ... | ... | ... |
| | 75 | 9.14 | 12.33 | 13.79 | 14.67 | 15.28 | 16.36 | 17.25 | ... | ... | ... | ... | ... | ... | ... |
| | 90 | 3.45 | 7.00 | 9.00 | 10.31 | 11.25 | 13.00 | 14.50 | ... | ... | ... | ... | ... | ... | ... |
| | 95 | 1.70 | 4.73 | 6.75 | 8.15 | 9.20 | 11.20 | 12.99 | ... | ... | ... | ... | ... | ... | ... |
| | 98 | 0.67 | 2.88 | 4.72 | 6.12 | 7.20 | 9.39 | 11.41 | ... | ... | ... | ... | ... | ... | ... |
| | 99 | 0.33 | 2.00 | 3.65 | 4.98 | 6.05 | 8.29 | 10.42 | ... | ... | ... | ... | ... | ... | ... |
| | 99.9 | 0.03 | 0.61 | 1.60 | 2.62 | 3.54 | 5.69 | 7.97 | ... | ... | ... | ... | ... | ... | ... |
| 4... | 50 | 38.57 | 38.57 | 38.57 | 38.57 | 38.57 | 38.57 | 38.57 | 15.91 | 15.91 | 15.91 | 15.91 | 15.91 | 15.91 | 15.91 |
| | 75 | 24.30 | 28.75 | 30.63 | 31.72 | 32.46 | 33.76 | 34.80 | 6.94 | 9.40 | 10.53 | 11.22 | 11.69 | 12.54 | 13.24 |
| | 90 | 14.22 | 20.96 | 24.09 | 25.99 | 27.29 | 29.61 | 31.51 | 2.60 | 5.30 | 6.83 | 7.83 | 8.56 | 9.92 | 11.09 |
| | 95 | 9.76 | 16.90 | 20.54 | 22.80 | 24.38 | 27.23 | 29.60 | 1.30 | 3.56 | 5.11 | 6.18 | 6.97 | 8.52 | 9.91 |
| | 98 | 6.01 | 12.94 | 16.90 | 19.46 | 21.29 | 24.66 | 27.51 | 0.51 | 2.16 | 3.56 | 4.62 | 5.45 | 7.12 | 8.68 |
| | 99 | 4.18 | 10.65 | 14.70 | 17.39 | 19.35 | 23.01 | 26.15 | 0.25 | 1.50 | 2.74 | 3.76 | 4.58 | 6.28 | 7.93 |
| | 99.9 | 1.31 | 5.75 | 9.50 | 12.30 | 14.45 | 18.69 | 22.51 | 0.03 | 0.46 | 1.21 | 1.97 | 2.67 | 4.29 | 6.03 |
| 5... | 50 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | 50.00 | 31.38 | 31.38 | 31.38 | 31.38 | 31.38 | 31.38 | 31.38 |
| | 75 | 35.94 | 40.51 | 42.38 | 43.46 | 44.18 | 45.44 | 46.44 | 19.38 | 23.06 | 24.63 | 25.55 | 26.17 | 27.26 | 28.15 |
| | 90 | 24.66 | 32.36 | 35.73 | 37.70 | 39.04 | 41.37 | 43.25 | 11.21 | 16.64 | 19.21 | 20.77 | 21.84 | 23.78 | 25.37 |
| | 95 | 18.93 | 27.81 | 31.91 | 34.35 | 36.03 | 38.98 | 41.37 | 7.64 | 13.35 | 16.30 | 18.14 | 19.44 | 21.80 | 23.77 |
| | 98 | 13.53 | 23.04 | 27.80 | 30.71 | 32.73 | 36.32 | 39.26 | 4.69 | 10.17 | 13.35 | 15.42 | 16.90 | 19.67 | 22.03 |
| | 99 | 10.56 | 20.12 | 25.19 | 28.37 | 30.58 | 34.58 | 37.87 | 3.27 | 8.35 | 11.58 | 13.75 | 15.33 | 18.31 | 20.90 |
| | 99.9 | 4.76 | 13.11 | 18.57 | 22.25 | 24.90 | 29.85 | 34.05 | 1.02 | 4.48 | 7.43 | 9.66 | 11.38 | 14.80 | 17.91 |
| 6... | 50 | 57.85 | 57.85 | 57.85 | 57.85 | 57.85 | 57.85 | 57.85 | 42.14 | 42.14 | 42.14 | 42.14 | 42.14 | 42.14 | 42.14 |
| | 75 | 44.68 | 49.06 | 50.82 | 51.84 | 52.51 | 53.67 | 54.60 | 29.71 | 33.67 | 35.32 | 36.27 | 36.91 | 38.03 | 38.93 |
| | 90 | 33.32 | 41.17 | 44.47 | 46.38 | 47.65 | 49.88 | 51.65 | 20.09 | 26.61 | 29.50 | 31.22 | 32.38 | 34.43 | 36.09 |
| | 95 | 27.13 | 36.58 | 40.71 | 43.13 | 44.76 | 47.60 | 49.87 | 15.32 | 22.73 | 26.22 | 28.32 | 29.76 | 32.33 | 34.42 |
| | 98 | 20.93 | 31.61 | 36.57 | 39.52 | 41.53 | 45.04 | 47.87 | 10.88 | 18.74 | 22.73 | 25.20 | 26.92 | 30.02 | 32.57 |
| | 99 | 17.31 | 28.45 | 33.88 | 37.15 | 39.39 | 43.35 | 46.54 | 8.47 | 16.31 | 20.54 | 23.21 | 25.09 | 28.51 | 31.36 |
| | 99.9 | 9.40 | 20.44 | 26.74 | 30.75 | 33.57 | 38.65 | 42.83 | 3.79 | 10.54 | 15.02 | 18.07 | 20.29 | 24.47 | 28.06 |
| 10... | 50 | 74.14 | 74.14 | 74.14 | 74.14 | 74.14 | 74.14 | 74.14 | 64.49 | 64.49 | 64.49 | 64.49 | 64.49 | 64.49 | 64.49 |
| | 75 | 64.46 | 67.80 | 69.11 | 69.85 | 70.33 | 71.18 | 71.84 | 54.23 | 57.69 | 59.07 | 59.86 | 60.38 | 61.28 | 61.99 |
| | 90 | 55.04 | 61.67 | 64.29 | 65.76 | 66.74 | 68.41 | 69.71 | 44.83 | 51.40 | 54.06 | 55.58 | 56.59 | 58.34 | 59.71 |
| | 95 | 49.31 | 57.87 | 61.30 | 63.24 | 64.52 | 66.70 | 68.40 | 39.33 | 47.60 | 51.02 | 52.98 | 54.29 | 56.55 | 58.33 |
| | 98 | 42.95 | 53.51 | 57.86 | 60.33 | 61.95 | 64.74 | 66.90 | 33.43 | 43.34 | 47.59 | 50.05 | 51.68 | 54.52 | 56.76 |
| | 99 | 38.82 | 50.58 | 55.53 | 58.35 | 60.21 | 63.41 | 65.89 | 29.71 | 40.54 | 45.30 | 48.08 | 49.94 | 53.16 | 55.71 |
| | 99.9 | 28.15 | 42.41 | 48.92 | 52.73 | 55.26 | 59.61 | 63.00 | 20.46 | 32.94 | 38.98 | 42.59 | 45.04 | 49.33 | 52.74 |
| 20... | 50 | 86.84 | 86.84 | 86.84 | 86.84 | 86.84 | 86.84 | 86.84 | 81.94 | 81.94 | 81.94 | 81.94 | 81.94 | 81.94 | 81.94 |
| | 75 | 81.33 | 83.27 | 84.03 | 84.45 | 84.73 | 85.20 | 85.58 | 75.79 | 77.93 | 78.76 | 79.23 | 79.55 | 80.08 | 80.50 |
| | 90 | 75.52 | 79.66 | 81.22 | 82.10 | 82.66 | 83.62 | 84.37 | 69.58 | 73.98 | 75.68 | 76.63 | 77.26 | 78.32 | 79.14 |
| | 95 | 71.74 | 77.31 | 79.43 | 80.60 | 81.36 | 82.64 | 83.62 | 65.63 | 71.48 | 73.74 | 75.00 | 75.83 | 77.23 | 78.31 |
| | 98 | 67.26 | 74.53 | 77.31 | 78.84 | 79.83 | 81.49 | 82.76 | 61.04 | 68.54 | 71.47 | 73.10 | 74.17 | 75.97 | 77.36 |
| | 99 | 64.17 | 72.59 | 75.83 | 77.62 | 78.77 | 80.70 | 82.17 | 57.93 | 66.52 | 69.91 | 71.80 | 73.03 | 75.11 | 76.71 |
| | 99.9 | 55.26 | 66.86 | 71.48 | 74.02 | 75.66 | 78.40 | 80.45 | 49.13 | 60.64 | 65.36 | 68.00 | 69.73 | 72.63 | 74.85 |

NOTE.—In evaluating fatigue curves faired through test data, subtract 3 from number of groups before entering table.

cal example, the risk in saying that at least half the population sampled will lie above the second-ranking point in five is

$$\begin{aligned} p &= 0.5^5 + 5x0.5^4(1 - 0.5) \\ &= 0.031 + 0.156 \\ &= 0.187 \end{aligned}$$

It follows that the confidence level in making this prediction is 81.3 per cent.

Extension of the reasoning to the $i$-th value obtained shows that the risk in a given "exceedance" prediction, $A_x$, is given by the first $i$ terms of the binomial expansion of

$$[A_x + (1 - A_x)]^n$$

The confidence level is the difference between this sum and unity.

*Analysis of Multiple Groups of n Tests:*

The next case to consider is that in which there have been tested $m$ groups of $n$ specimens each, and only the lowest value in each group has been recorded. If $(m + 1)/2$ or more of these "least-of-$n$" values exceed $x$, then the median of the $m$ values, being of necessity one of the exceeding points, is some number[3] greater than $x$. It follows that some proportion less than $A_x$ of the population sampled will exceed the median, or middlemost, of the "least-of-$n$" values we have obtained.

Previously, in dealing with $n$ specimens only, we noted that the probability for any one of them to exceed $x$ was $A_x$, and for $n$ consecutive values to exceed $x$, $A_x^n$. The latter value is also the individual probability for a "least-of-$n$" value to exceed $x$. Except for the addition of the exponent $n$, the problem of multiple groups now becomes identical with that dealt with in considering the $i$-th ranking point in a group. Thus it can be said that in $m$ groups, the probability, or risk, of having $(m + 1)/2$ or more exceed $x$ is given by the sum of the first $(m + 1)/2$ terms in the binomial expansion of

$$[A_x^n + (1 - A_x^n)]^m$$

The final complication is to assume that there have been determined the $i$ lowest values from each of $m$ groups of $n$ specimens. We now determine the median value for the $i$-th ranking points from each of the groups. What, then, is the risk that a proportion less than $A_x$ of the population exceeds the median so determined? (The confidence that $A_x$ or more will exceed proceeds automatically from the answer to this question, since it is simply one minus the risk.)

This question may be answered with a substitution similar to that just employed. Now, however, instead of substituting $A_x^n$ for $A_x$, it is necessary to substitute the more complex probability function for the $i$-th ranking value in a group, which for the moment will be called $Q$. With this terminology, the risk that less than $A_x$ will exceed the median of the $i$-th ranking values from the several groups is given by the sum of the first $(m + 1)/2$ terms of the binomial expansion of

$$[Q + (1 - Q)]^m$$

where:

$Q$ is the sum of the first $i$ terms of the expansion of

$$[A_x + (1 - A_x)]^n.$$

This completes the necessary analytical development to permit the calculation of corresponding values of "exceedances" and confidence levels for median values of points of any desired rank in any given number of groups of any fixed number of specimens. An extensive series of calculations was carried out on electronic calculators, and the resulting values are listed in Table I.

---

[3] To avoid unnecessary complications, $m$ is assumed to be always an odd integer; in practical use, it will be adequate to interpolate for even values of $m$.

It is perhaps desirable to point out that for the determination of a single variable under constant test conditions, which is the problem considered so far, the development is not only completely rigorous, but also completely independent of the nature of the frequency distribution for the population being sampled.

As an example of the use of Table I for a problem of this type, let us suppose that life tests are routinely conducted on an item of electrical equipment. The facilities permit testing in groups of six, and the tests are stopped as soon as two of the test items have failed. After seven such group tests, the recorded times to first failure are 37, 42, 36, 50, 47, 41, and 34 hr; times to the second failure in each group are 51, 44, 39, 51, 57, 48, and 49 hr. The median of the lowest ranking points (first failures) from the seven groups is 41 hr; second ranking points (second failures), 49 hr. Entering Table I for six points per group and seven groups, we note in the column for "lowest ranking points" that the following predictions can be made; with a confidence level of 50 per cent, at least 89.1 per cent of the product can be expected to give a life greater than 41 hr; with a confidence level of 75 per cent, at least 85.1 per cent can be expected to exceed this life; with a confidence level of 90 per cent, at least 80.8 per cent; etc. Likewise, from the column for "second ranking points," with a confidence level of 50 per cent, at least 73.6 per cent can be expected to exceed 49 hr; for 75 per cent confidence, at least 68.0 per cent; for 90 per cent confidence, at least 62.7 per cent; etc.

## PROBABILITY ANALYSIS IN CURVE DETERMINATIONS

Up to this point, complete mathematical rigor has been retained in the analysis of the problem. In determining a curve of a given test result *versus* a variable test condition, it is probably feasible to continue the rigorous development if the equation of the sought-after curve is known and only its constants need determination. This situation, however, is rare in practice. When it does exist, the data are susceptible to least-squares analysis, a method with which the author has no quarrel.

The more common situation is the one in which only the general qualitative nature of the curve is predictable, while the factors governing its precise mathematical shape are indeterminate. For this situation, the experimenter's intuition is often a more reliable guide than any amount of mathematics, however rigorous.

The approach to the problem in any given case should be governed by the degree of "unknownness" in the curve. For this reason, only a single type of curve is dealt with here; the procedure outlined is intended to serve only as a guide to those dealing with other types of experimental relationship.

On the basis of primary interest to the author, the example chosen is that of the *S-N* curve in fatigue testing. This curve is known to have at all points a negative slope, and is concave upward over a major portion of its length. (The short-life end of the curve is presumably concave downward, but it has only recently been the subject of serious investigation and is ignored for purposes of the present development.)

It might be noted in passing that a number of equations for the *S-N* curve have been proposed, but none has been accompanied by any proof of its phenomenological validity. In general, they have served as a convenience in plotting rather than as a definition of the phenomena involved.

Without an equation for the curve, our intuition tells us that four points must be determined in order to yield a first approximation to its shape; for, while three points may define direction of curvature, there remains infinite freedom in locating the region or regions of maximum curvature. In the single-value problem, the first point obtained is a first approximation; in the *S-N* problem, the first four points provide the first approximation. In either case, subsequent points serve to improve the accuracy of the approximation. Assuming that engineering judgment born of some experience in the field is available to assist, then it is not unreasonable to say that the degree of refinement provided by additional points after the first approximation is the same in both the single-point and the curve problems. Thus, for the curve problem *versus* the single-point problem, four points are equivalent to one, five to two, six to three, etc. This reasoning accounts for the note appearing at the bottom of Table I.[4]

TABLE II.—RANK FOR EQUIVALENT SIGNIFICANCE IN GROUPS OF DIFFERENT SIZES.

| Number of Tests in Group | Rank of Test Value for Indicated Values of Confidence/ "Exceedance," per cent | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 75/ 63.0 | 75/ 70.7 | 75/ 75.8 | 75/ 79.4 | 75/ 82.0 | 75/ 84.1 | 75/ 85.7 | 75/ 87.1 | 95/ 36.8 | 95/ 47.3 | 95/ 54.9 | 95/ 60.7 | 95/ 65.2 | 95/ 68.8 | 95/ 71.7 | 95/ 74.1 |
| 3 | 1.0 | ... | ... | ... | ... | ... | ... | ... | 1.0 | ... | ... | ... | ... | ... | ... | ... |
| 4 | 1.3 | 1.0 | ... | ... | ... | ... | ... | ... | 1.4 | 1.0 | ... | ... | ... | ... | ... | ... |
| 5 | 1.6 | 1.2 | 1.0 | ... | ... | ... | ... | ... | 1.9 | 1.4 | 1.0 | ... | ... | ... | ... | ... |
| 6 | 1.9 | 1.5 | 1.2 | 1.0 | ... | ... | ... | ... | 2.3 | 1.7 | 1.3 | 1.0 | ... | ... | ... | ... |
| 7 | 2.2 | 1.7 | 1.4 | 1.2 | 1.0 | ... | ... | ... | 2.8 | 2.1 | 1.6 | 1.2 | 1.0 | ... | ... | ... |
| 8 | 2.5 | 1.9 | 1.6 | 1.3 | 1.2 | 1.0 | ... | ... | 3.3 | 2.5 | 1.9 | 1.5 | 1.2 | 1.0 | ... | ... |
| 9 | 2.8 | 2.2 | 1.8 | 1.5 | 1.3 | 1.1 | 1.0 | ... | 3.8 | 2.8 | 2.2 | 1.7 | 1.4 | 1.2 | 1.0 | ... |
| 10 | 3.1 | 2.4 | 2.0 | 1.6 | 1.5 | 1.3 | 1.1 | 1.0 | 4.2 | 3.2 | 2.5 | 2.0 | 1.7 | 1.4 | 1.2 | 1.0 |
| 11 | 3.5 | 2.6 | 2.1 | 1.8 | 1.6 | 1.4 | 1.2 | 1.1 | 4.8 | 3.6 | 2.8 | 2.3 | 1.9 | 1.6 | 1.3 | 1.1 |
| 12 | 3.8 | 2.9 | 2.3 | 2.0 | 1.7 | 1.5 | 1.3 | 1.2 | 5.3 | 4.0 | 3.1 | 2.5 | 2.1 | 1.8 | 1.5 | 1.3 |
| 13 | 4.1 | 3.1 | 2.5 | 2.1 | 1.9 | 1.6 | 1.4 | 1.3 | 5.8 | 4.4 | 3.4 | 2.8 | 2.3 | 2.0 | 1.6 | 1.4 |
| 14 | 4.5 | 3.4 | 2.7 | 2.3 | 2.0 | 1.7 | 1.5 | 1.4 | 6.4 | 4.8 | 3.8 | 3.1 | 2.5 | 2.2 | 1.8 | 1.6 |
| 15 | 4.8 | 3.7 | 2.9 | 2.5 | 2.1 | 1.8 | 1.6 | 1.5 | 6.9 | 5.2 | 4.1 | 3.4 | 2.8 | 2.4 | 2.0 | 1.8 |
| 16 | 5.1 | 3.9 | 3.2 | 2.6 | 2.3 | 2.0 | 1.8 | 1.6 | 7.4 | 5.6 | 4.5 | 3.7 | 3.0 | 2.6 | 2.2 | 1.9 |
| 18 | 5.8 | 4.4 | 3.6 | 3.0 | 2.5 | 2.2 | 2.0 | 1.8 | 8.5 | 6.5 | 5.2 | 4.3 | 3.6 | 3.1 | 2.6 | 2.3 |
| 20 | 6.5 | 5.0 | 4.0 | 3.3 | 2.8 | 2.5 | 2.2 | 2.0 | 9.5 | 7.4 | 5.9 | 4.9 | 4.1 | 3.5 | 3.0 | 2.6 |
| 25 | 8.1 | 6.3 | 5.1 | 4.2 | 3.6 | 3.1 | 2.8 | 2.5 | 12.3 | 9.6 | 7.8 | 6.4 | 5.5 | 4.7 | 4.1 | 3.6 |
| 30 | 9.8 | 7.6 | 6.2 | 5.1 | 4.4 | 3.8 | 3.4 | 3.1 | 15.1 | 11.9 | 9.7 | 8.0 | 6.8 | 5.9 | 5.1 | 4.6 |
| 35 | 11.3 | 8.9 | 7.2 | 6.0 | 5.2 | 4.5 | 4.0 | 3.6 | 17.9 | 14.1 | 11.5 | 9.6 | 8.2 | 7.1 | 6.2 | 5.5 |
| 40 | 12.7 | 10.2 | 8.3 | 6.9 | 6.0 | 5.2 | 4.6 | 4.1 | 20.7 | 16.4 | 13.4 | 11.2 | 9.6 | 8.3 | 7.3 | 6.5 |
| 50 | 15.7 | 12.8 | 10.5 | 8.7 | 7.6 | 6.5 | 5.8 | 5.2 | 26.3 | 20.9 | 17.2 | 14.3 | 12.3 | 10.7 | 9.5 | 8.4 |

## Simplified Handling of Simple Problems

A problem frequently encountered in the necessarily accelerated testing common to industrial laboratories is that of comparing several products on the basis of a few tests of each. Often, a different number of tests is made on each of the products, and a valid comparison without some knowledge of the governing probabilities is difficult at best. The methods of this paper can be effectively used in dealing with this situation.

A frequent approach to the reporting of data of this type is to list the average and the minimum value obtained for each of the products, along with the number of tests conducted in each case. Further analysis is of a nonformalized nature, and depends almost entirely on human judgment. A valuable assistance to judgment would be provided if the several products could be compared at a level of equal probability.

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[4] The procedure of subtracting 3 from the number of points before entering the table admittedly has no objective foundation. It represents only the author's suggestion for a simple and practical approach and should not be treated as having any rigorous justification.

Table I listed the corresponding confidence levels and exceedances for the first four ranking points from groups of several sizes; these are the entries for the single group ($m = 1$).

If the calculations are extended to include points of higher rank than fourth and additional group sizes, then interpolation will show at what rank in a group of a given size a given confidence-"exceedance" combination may be found. The use of interpolation is an approximation involving certain nonrigorous assumptions regarding the nature of the frequency distribution, but the inaccuracies thus introduced are of a totally insignificant magnitude for the type of problem being considered.

The values of rank obtained by the interpolation are listed in Table II for a number of group sizes. The specific confidence-"exceedance" combinations chosen for listing in this table are those corresponding to the lowest ranking points in groups of from three to ten specimens. An example of the use of this table is given subsequent to the introduction of Table III, which follows below.

It is possible that a slightly more refined analysis of comparative test data may be desired. The value corresponding to any given "exceedance", at a fixed confidence level, can be obtained by using the "exceedance" values for the individual points as plotting points on a probability graph. Table III lists "exceedance" values calculated for use in this manner. The manner in which this table is used will be made clear in the example that follows.

*Example (Tables II and III):*

Let us suppose that three materials have been tested for use in an application where breaking load is the criterion of performance. The number of tests was limited by the quantity of each material at hand. Results of the hypothetical tests were as follows:

BREAKING LOAD, lb.

| Material A | | Material B | Material C | | |
|---|---|---|---|---|---|
| 385 | 396 | 389 | 382 | 369 | 376 |
| 358 | 363 | 400 | 396 | 401 | 386 |
| 380 | 393 | 359 | 356 | 361 | 378 |
| 374 | 403 | 372 | 376 | 356 | 387 |
| 369 | 374 | 379 | 374 | 395 | 374 |
| 385 | | | 368 | 391 | 385 |
| | | | 381 | | |
| Avg. 380.0<br>Min. 358<br>11 tests | | Avg. 379.8<br>Min. 359<br>5 tests | Avg. 379.0<br>Min. 356<br>19 tests | | |

Simple observation of the averages and minima, without consideration for the number of tests run in each case, would indicate that material B is the best of the three. Since material B was the least extensively explored, however, it is clear that the data cannot be considered as conclusive for this material as for the others. Reference to Table II will show just how much the difference in numbers affects the soundness of the choice. Since the smallest number in any group was five, let us consider the significance of the minimum in this case, and compare the other materials at the same level of significance.

It will be noted in Table II that we can be 95 per cent confident that at least 54.9 per cent of material B will exceed the minimum obtained in the five tests. (The sixth column from the right shows that this confidence-"exceedance" combination corresponds to the point of rank 1 in a group of five.) Retaining these values of confidence and "exceedance," we note that in a group of 11 tests, the equivalent rank is 2.8, while in a group of 19 tests, it is, by interpolation, 5.6. To find the strength levels corresponding to these fictitious ranks in each group,

TABLE III.—"EXCEEDANCE" PLOTTING POINTS PERCENTAGE FOR ANALYSIS OF GROUPS USING PROBABILITY PAPER.

| Rank of Test Point | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 75 per cent Confidence | | | | | | | | | | | | | | | | |
| Number of tests: | | | | | | | | | | | | | | | | |
| 3 | 63.0 | 32.6 | 9.14 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 4 | 70.7 | 45.6 | 24.3 | 6.94 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 5 | 75.8 | 54.6 | 35.9 | 19.4 | 5.59 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 6 | 79.4 | 61.1 | 44.7 | 29.7 | 16.1 | 4.68 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 7 | 82.0 | 65.9 | 51.3 | 37.9 | 25.3 | 13.8 | 4.03 | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 8 | 84.1 | 69.7 | 56.7 | 44.4 | 32.9 | 22.1 | 12.1 | 3.53 | ... | ... | ... | ... | ... | ... | ... | ... |
| 9 | 85.7 | 72.8 | 60.9 | 49.8 | 39.2 | 29.1 | 19.5 | 10.7 | 3.15 | ... | ... | ... | ... | ... | ... | ... |
| 10 | 87.1 | 75.3 | 64.5 | 54.2 | 44.4 | 35.1 | 26.1 | 17.6 | 9.64 | 2.84 | ... | ... | ... | ... | ... | ... |
| 11 | 88.2 | 77.3 | 67.4 | 58.0 | 48.9 | 40.2 | 31.7 | 23.6 | 15.9 | 8.76 | 2.58 | ... | ... | ... | ... | ... |
| 12 | 89.1 | 79.1 | 69.9 | 61.1 | 52.7 | 44.5 | 36.6 | 29.0 | 21.6 | 14.6 | 8.03 | 2.37 | ... | ... | ... | ... |
| 13 | 89.9 | 80.6 | 72.0 | 63.8 | 56.0 | 48.3 | 40.9 | 33.7 | 26.7 | 19.9 | 13.4 | 7.41 | 2.19 | ... | ... | ... |
| 14 | 90.57 | 81.9 | 73.9 | 66.2 | 58.8 | 51.6 | 44.6 | 37.8 | 31.2 | 24.7 | 18.5 | 12.5 | 6.88 | 2.03 | ... | ... |
| 15 | 91.17 | 83.0 | 75.5 | 68.3 | 61.3 | 54.6 | 48.0 | 41.5 | 35.2 | 29.0 | 23.0 | 17.2 | 11.6 | 6.42 | 1.90 | ... |
| 16 | 91.70 | 84.0 | 76.9 | 70.1 | 63.6 | 57.2 | 50.9 | 44.8 | 38.8 | 32.9 | 27.1 | 21.5 | 16.1 | 10.9 | 6.02 | 1.78 |
| 17 | 92.17 | 84.9 | 78.2 | 71.8 | 65.6 | 59.5 | 53.5 | 47.7 | 42.0 | 36.4 | 30.9 | 25.5 | 20.2 | 15.6 | 10.2 | 5.66 |
| 18 | 92.59 | 85.7 | 79.4 | 73.3 | 67.3 | 61.6 | 55.9 | 50.4 | 44.9 | 39.5 | 34.3 | 29.1 | 24.0 | 19.1 | 14.3 | 9.67 |
| 19 | 92.96 | 86.4 | 80.4 | 74.6 | 69.0 | 63.4 | 58.1 | 52.7 | 47.5 | 42.4 | 37.4 | 32.4 | 27.5 | 22.7 | 18.1 | 13.5 |
| 20 | 93.30 | 87.1 | 81.3 | 75.8 | 70.4 | 65.2 | 60.0 | 54.9 | 49.9 | 45.0 | 40.2 | 35.4 | 30.7 | 26.1 | 21.6 | 17.1 |
| 21 | 93.61 | 87.7 | 82.2 | 76.9 | 71.7 | 66.7 | 61.8 | 56.9 | 52.1 | 47.4 | 42.7 | 38.2 | 33.7 | 29.2 | 24.8 | 20.5 |
| 22 | 93.89 | 88.2 | 82.9 | 77.9 | 73.0 | 68.1 | 63.4 | 58.7 | 54.1 | 49.6 | 45.1 | 40.7 | 36.4 | 32.1 | 27.8 | 23.7 |
| 23 | 94.16 | 88.7 | 83.6 | 78.8 | 74.1 | 69.4 | 64.9 | 60.4 | 56.0 | 51.6 | 47.3 | 43.1 | 38.9 | 34.7 | 30.6 | 26.6 |
| 24 | 94.39 | 89.2 | 84.3 | 79.6 | 75.1 | 70.6 | 66.3 | 62.0 | 57.7 | 53.5 | 49.4 | 45.2 | 41.2 | 37.2 | 33.2 | 29.3 |
| 25 | 94.61 | 89.6 | 84.9 | 80.4 | 76.0 | 71.7 | 67.5 | 63.4 | 59.3 | 55.2 | 51.2 | 47.3 | 43.3 | 39.5 | 35.6 | 31.8 |

90 PER CENT CONFIDENCE

| Number of Tests: | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | 46.4 | 19.6 | 3.45 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 4 | 56.2 | 32.0 | 14.2 | 2.60 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 5 | 63.1 | 41.6 | 24.7 | 11.2 | 2.09 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 6 | 68.1 | 49.0 | 33.3 | 20.1 | 9.26 | 1.74 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 7 | 72.0 | 54.7 | 40.4 | 27.9 | 17.0 | 7.88 | 1.49 | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 8 | 75.0 | 59.4 | 46.2 | 34.5 | 24.0 | 14.7 | 6.86 | 1.31 | ... | ... | ... | ... | ... | ... | ... | ... |
| 9 | 77.4 | 63.2 | 51.0 | 40.1 | 30.1 | 21.0 | 12.9 | 6.07 | 1.16 | ... | ... | ... | ... | ... | ... | ... |
| 10 | 79.4 | 66.3 | 55.0 | 44.8 | 35.4 | 26.7 | 18.7 | 11.6 | 5.44 | 1.05 | ... | ... | ... | ... | ... | ... |
| 11 | 81.1 | 69.0 | 58.5 | 48.9 | 40.1 | 31.8 | 24.1 | 16.9 | 10.5 | 4.94 | 0.95 | ... | ... | ... | ... | ... |
| 12 | 82.5 | 71.2 | 61.4 | 52.5 | 44.1 | 36.2 | 28.8 | 21.9 | 15.4 | 9.55 | 4.51 | 0.87 | ... | ... | ... | ... |
| 13 | 83.8 | 73.2 | 64.0 | 55.6 | 47.6 | 40.2 | 33.1 | 26.4 | 20.0 | 14.2 | 8.79 | 4.16 | 0.81 | ... | ... | ... |
| 14 | 84.8 | 74.9 | 66.3 | 58.3 | 50.8 | 43.7 | 36.9 | 30.4 | 24.3 | 18.5 | 13.1 | 8.14 | 3.86 | 0.75 | ... | ... |
| 15 | 85.8 | 76.4 | 68.3 | 60.7 | 53.6 | 46.8 | 40.3 | 34.1 | 28.2 | 22.5 | 17.2 | 12.2 | 7.57 | 3.59 | 0.70 | ... |
| 16 | 86.6 | 77.8 | 70.0 | 62.9 | 56.1 | 49.6 | 43.4 | 37.5 | 31.8 | 26.3 | 21.0 | 16.1 | 11.4 | 7.09 | 3.36 | 0.66 |
| 17 | 87.3 | 79.0 | 71.6 | 64.8 | 58.3 | 52.2 | 46.3 | 40.5 | 35.0 | 29.7 | 24.6 | 19.7 | 15.1 | 10.7 | 6.65 | 3.17 |
| 18 | 88.0 | 80.0 | 73.1 | 66.5 | 60.4 | 54.5 | 48.8 | 43.3 | 38.0 | 32.9 | 27.9 | 23.1 | 18.5 | 14.2 | 10.1 | 6.27 |
| 19 | 88.6 | 81.0 | 74.3 | 68.1 | 62.2 | 56.6 | 51.1 | 45.9 | 40.7 | 35.8 | 31.0 | 26.3 | 21.8 | 17.5 | 13.4 | 9.50 |
| 20 | 89.1 | 81.9 | 75.5 | 69.6 | 63.9 | 58.5 | 53.3 | 48.2 | 43.3 | 38.5 | 33.8 | 29.3 | 24.9 | 20.7 | 16.6 | 12.7 |
| 21 | 89.6 | 82.7 | 76.6 | 70.9 | 65.5 | 60.3 | 55.2 | 50.3 | 45.6 | 41.0 | 36.4 | 32.0 | 27.8 | 23.6 | 19.6 | 15.7 |
| 22 | 90.06 | 83.4 | 77.6 | 72.1 | 66.9 | 61.9 | 57.0 | 52.3 | 47.7 | 43.2 | 48.9 | 34.6 | 30.4 | 26.4 | 22.5 | 18.7 |
| 23 | 90.47 | 84.1 | 78.5 | 73.2 | 68.2 | 63.4 | 58.7 | 54.1 | 49.7 | 45.4 | 41.1 | 37.0 | 33.0 | 29.0 | 25.2 | 21.4 |
| 24 | 90.85 | 84.7 | 79.3 | 74.2 | 69.4 | 64.7 | 60.2 | 55.8 | 51.5 | 47.3 | 43.2 | 39.2 | 35.3 | 31.5 | 27.7 | 24.1 |
| 25 | 91.20 | 85.3 | 80.1 | 75.2 | 70.5 | 66.0 | 61.7 | 57.4 | 53.3 | 49.2 | 45.2 | 41.3 | 37.5 | 33.8 | 30.1 | 26.5 |

(Continued on p. 1314)

## TABLE III.—*Concluded.*

95 PER CENT CONFIDENCE

| Number of Tests: | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | 36.8 | 13.5 | 1.70 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 4 | 47.3 | 24.9 | 9.76 | 1.30 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 5 | 54.9 | 34.3 | 18.9 | 7.64 | 1.02 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 6 | 60.7 | 41.8 | 27.1 | 15.3 | 6.27 | 0.85 | ... | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 7 | 65.2 | 48.0 | 34.1 | 22.5 | 12.9 | 5.32 | 0.73 | ... | ... | ... | ... | ... | ... | ... | ... | ... |
| 8 | 68.8 | 52.9 | 40.2 | 28.9 | 19.3 | 11.1 | 4.62 | 0.64 | ... | ... | ... | ... | ... | ... | ... | ... |
| 9 | 71.7 | 57.1 | 45.0 | 34.5 | 25.1 | 16.9 | 9.76 | 4.10 | 0.57 | ... | ... | ... | ... | ... | ... | ... |
| 10 | 74.1 | 60.6 | 49.3 | 39.3 | 30.3 | 22.2 | 15.0 | 8.71 | 3.66 | 0.51 | ... | ... | ... | ... | ... | ... |
| 11 | 76.2 | 63.6 | 53.0 | 43.5 | 35.0 | 27.1 | 20.0 | 13.5 | 7.87 | 3.31 | 0.47 | ... | ... | ... | ... | ... |
| 12 | 77.9 | 66.1 | 56.2 | 47.3 | 39.1 | 31.5 | 24.5 | 18.1 | 12.3 | 7.17 | 3.04 | 0.43 | ... | ... | ... | ... |
| 13 | 79.4 | 68.4 | 59.0 | 50.5 | 42.7 | 35.5 | 28.7 | 22.4 | 16.5 | 11.3 | 6.58 | 2.79 | 0.39 | ... | ... | ... |
| 14 | 80.7 | 70.3 | 61.4 | 53.4 | 46.0 | 39.0 | 32.5 | 26.3 | 20.6 | 15.3 | 10.4 | 6.10 | 2.57 | 0.37 | ... | ... |
| 15 | 81.9 | 72.1 | 63.6 | 56.0 | 48.9 | 42.2 | 36.0 | 30.0 | 24.4 | 19.1 | 14.2 | 9.15 | 5.66 | 2.39 | 0.34 | ... |
| 16 | 82.9 | 73.6 | 65.6 | 58.3 | 51.6 | 45.2 | 39.1 | 33.3 | 27.9 | 22.7 | 17.8 | 13.2 | 9.02 | 5.29 | 2.24 | 0.32 |
| 17 | 83.8 | 75.0 | 67.4 | 60.4 | 53.9 | 47.8 | 42.0 | 36.4 | 31.1 | 26.0 | 21.2 | 16.6 | 12.8 | 8.44 | 4.99 | 2.12 |
| 18 | 84.7 | 76.2 | 69.0 | 62.3 | 56.1 | 50.2 | 44.6 | 39.2 | 34.1 | 29.1 | 24.4 | 19.9 | 15.6 | 11.6 | 7.97 | 4.67 |
| 19 | 85.4 | 77.3 | 70.4 | 64.1 | 58.1 | 52.4 | 47.0 | 41.8 | 36.8 | 32.0 | 27.4 | 23.0 | 18.7 | 14.7 | 11.0 | 7.50 |
| 20 | 86.1 | 78.4 | 71.7 | 65.6 | 59.9 | 54.4 | 49.2 | 44.2 | 39.3 | 34.7 | 30.2 | 25.9 | 21.7 | 17.7 | 14.0 | 10.4 |
| 21 | 86.7 | 79.3 | 72.9 | 67.1 | 61.5 | 56.3 | 51.2 | 46.4 | 41.7 | 37.2 | 32.8 | 28.6 | 24.5 | 20.6 | 16.8 | 13.2 |
| 22 | 87.3 | 80.2 | 74.0 | 68.4 | 63.1 | 58.0 | 53.1 | 48.4 | 43.9 | 39.5 | 35.2 | 31.1 | 27.1 | 23.3 | 19.5 | 16.0 |
| 23 | 87.8 | 81.0 | 75.1 | 69.6 | 64.5 | 59.6 | 54.9 | 50.3 | 46.0 | 41.7 | 37.5 | 33.5 | 29.6 | 25.8 | 22.2 | 18.6 |
| 24 | 88.3 | 81.7 | 76.0 | 70.8 | 65.8 | 61.1 | 56.5 | 52.1 | 47.8 | 43.7 | 39.7 | 35.7 | 31.9 | 28.2 | 24.6 | 21.1 |
| 25 | 88.7 | 82.4 | 76.9 | 71.8 | 67.0 | 62.4 | 58.0 | 53.8 | 49.6 | 45.6 | 41.7 | 37.9 | 34.1 | 30.5 | 27.0 | 23.5 |

we must first regroup the test results in order of rank, from the lowest to the highest, as follows:

| Material A | | Material B | Material C | | |
|---|---|---|---|---|---|
| 358 | 385 | 359 | 356 | 376 | 386 |
| 363 | 385 | 372 | 361 | 376 | 387 |
| 369 | 393 | 379 | 365 | 378 | 391 |
| 374 | 396 | 389 | 368 | 381 | 395 |
| 374 | 403 | 400 | 369 | 382 | 396 |
| 380 | | | 374 | 385 | 401 |
| | | | 374 | | |

Resorting again to interpolation, we find, for each material, the strength level that can be expected, with 95 per cent confidence, to be exceeded by 54.9 per cent of the product:

Material A—Rank 2.8 .............. 367.8 lb
Material B—Rank 1.0 .............. 359.0 lb
Material C—Rank 5.6 .............. 372.0 lb

On the basis of these comparisons at equivalent levels, we find the choice going to material C, with the previous choice, material B, coming in last.

The procedure is seen to be quite simple, and requires no foreknowledge of statistical methods to put it into practice; it should be useful in a wide variety of data-handling problems.

A more refined approach, which may be useful in special cases, is provided by using probability graph paper. It will be noted in the third block of Table III (95 per cent confidence) that the plotting points for the data from five tests, ranked in order from the lowest to the highest value, are 54.9, 34.3, 18.9, 7.64, and 1.02, respectively. These are the percentages that may be expected, with 95 per cent confidence, to exceed the strength levels obtained in each of the five tests. Likewise, with 11 tests, the plotting points in order of rank are 76.2, 63.6, 53.0, etc. Ranking the test results as before and using the listed plotting points, we obtain the three curves in Fig. 2. As an example, we can pick from these curves the strength that can be expected to be exceeded by 60 per cent of the product, at a confidence level of 95 per cent. These are listed on the figure; it is not surprising that material C again emerges as the best choice.

It might be noted here that it is not essential to use probability graph paper in making the plots; its use, however, does have the effect of smoothing the curves and facilitating comparisons. The use of proba-

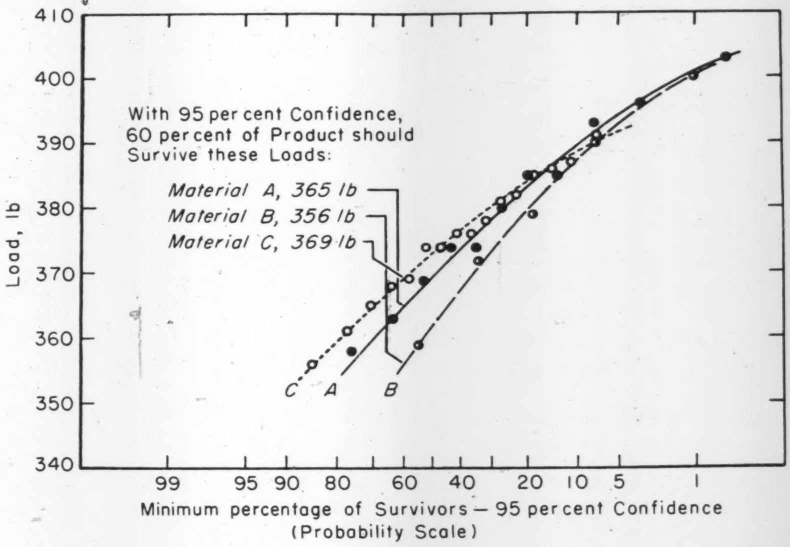

FIG. 2.—Comparative "Exceedances" for Three Materials at a Confidence Level of 95 per cent

bility paper *should not* be expected to result in straight-line plots; the points themselves are the only reliable indication of the proper curve shape.[5]

## CONCLUDING DISCUSSION

The methods presented herein are not intended to substitute for good engineering judgment, but only to provide it with a useful assist. It should be borne in mind that probability and statistical theory can only reveal how closely a given statement approaches certainty—it cannot create certainty. In many instances, it may serve only to reveal how uncertain one is about something that may have been thought to be proven fact.

Statements made regarding the significance of a given set of data may be at variance, when based on the principles here presented, from those that would result from an application of classical statistical theory. In general, classical theory will show higher confidence levels for similar predictions than will be obtained by the methods given here. The difference lies in the nonparametric nature of these methods. That is, they involve no assumptions whatsoever regarding the nature of the frequency distribution of the variable under consideration. The validity of confidence levels based on an assumption of, say, a statistically normal distribution, rests on the validity of that assumption. If there is limited confidence that the distribution is as assumed, then it is patently useless to calculate an answer to a confidence level of 99.9 per cent. It is this kind of situation for which the present methods are intended to be used.

Only experience will indicate the extent to which the tables given in this paper can be utilized, or the extent to which they should be expanded. Should certain extensions prove useful before they have become available, it is possible to derive approximate values of the type given in Table I by reference to tables published by the National Bureau of Standards.[6] For convenience, the necessary conversions in nomenclature are given here:

| | NBS Tables | This Paper |
|---|---|---|
| "Exceedance" $\geq 0.5$ (50 per cent)..... | $p$ | 1 − "exceedance" |
| | $n$ | $n$ |
| | $r$ | rank |
| | $\Sigma$ | confidence |
| "Exceedance" $\leq 0.5$ (50 per cent)..... | $p$ | "exceedance" |
| | $n$ | $n$ |
| | $r$ | $n - 1 -$ rank |
| | $\Sigma$ | risk (1 − confidence) |

The tables are useful for obtaining values pertinent to single groups ($m = 1$). For the additional calculations relating to multiple groups of tests, there is no tabular shortcut known to the author.

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[5] It is a common misconception that points obtained by random sampling from a normal distribution will always result in a straight line on probability paper; this is a fortuitous result only, unless the line represents the complete infinity of possible values.

[6] National Bureau of Standards, "Tables of Binomial Probability Distribution," U. S. Government Printing Office (1950).

# DISCUSSION

Mr. George Shombert, Jr.[1]—In the example cited by the author, materials A, B, and C test so much alike that the usual statistical methods will not distinguish between them. The variances are homogeneous, and the means are not significantly different. Yet the method described by the author indicates a clean-cut preference for material C.

If material C is actually better than A and B, then this method is extremely sensitive and represents a valuable addition to the engineer's kit of tools. However, if material C is not different from the others, a method which finds a difference where none exists is not likely to be useful to engineers, however handy it may be to salesmen.

Since it is stated that no more test specimens are available, we cannot resort to the obvious method of doing additional testing to determine whether material C is actually any better than A and B. Perhaps we can make the present data answer the question by arranging the numbers a little differently.

Suppose that before material C was tested, the samples had been divided into 3 groups and re-labelled D, E, and F. This of course, might have happened in any one of a large number of ways; specifically let us assume that it happened in the following way:

| | Material D | Material E | Material F |
|---|---|---|---|
| | 382 | 401 | 356 |
| | 374 | 376 | 368 |
| | 381 | 369 | 396 |
| | 361 | 365 | 385 |
| | 391 | 376 | 374 |
| | 378 | 387 | 395 |
| | 386 | | |
| Average Value | 379 | 379 | 379 |
| Variance | 93 | 172 | 209 |
| Minimum Value | 361 | 365 | 356 |
| Next Lowest Value | 374 | 369 | 368 |
| Rank for 95 to 54.9 per cent | 1.6 | 1.3 | 1.3 |
| Value for 95 to 54.9 per cent | 368.8 | 366.2 | 359.6 |

If we now rate the 5 materials A, B, D, E, and F according to the value calculated for 95 per cent confidence of 54.9 per cent "exceedance," we get:

| | |
|---|---|
| Material D | 368.8 |
| Material A | 367.8 |
| Material E | 366.2 |
| Material F | 359.6 |
| Material B | 359.0 |

Thus it can be seen that the author's method makes a distinction where there is no difference, since materials D, E, and F are all actually the same. Needless to say, conventional statistical theory will indicate that the pseudo-materials D, E, and F are the same, since they have the same average value and the variances are not significantly different (by either F-ratio or Cochran's test).

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[1] Assistant to Works Manager, Allis-Chalmers Manufacturing Co., Milwaukee, Wis.

The basic trouble seems to be that this method does not make use of all the available information. Only the values at the lower end of the distribution curve are used. On the other hand, the calculations for average value and standard deviation use all the data.

One can, of course, sympathize with the desire to reduce or eliminate the labor of arithmetical computation. However, several resources are available. For example, coding of the data will usually result in a saving of time and effort. Estimation of the standard deviation from the range will give results accurate enough for many purposes.

If it is desired to avoid the assumption of normality in interpretation of the data, one may either:

(*a*) use a more suitable distribution if sufficient knowledge exists for selection, or

(*b*) make use of Tchebycheff's inequality which makes no assumption whatever as to distribution.

MISS MARY TORREY.[2]—In the author's paper he gave another analysis of the data in which he plotted the values of their ranks on normal probability paper. The reviewer discovered that if you just plot the actual data, you could reach the same conclusion, and that is a much simpler procedure. The only trouble with either method is that there appears to be no way to compute the significance of the observed differences. Using the classical F test to compare the variances of materials B and C, shows that they are not significantly different even at the 10 per cent level of significance. There are several nonparametric tests that may be used. One of these also indicates that materials C and B probably come from the same population, and that probability is large, greater than 20 per cent.

With regard to using the median of minimum values if there are several samples, has the author explored the possibility of pooling the data from several samples and using some order statistic of the pooled data?

The second test I mentioned, which indicated a probability of greater than 20 per cent, makes the same assumption that the author's test does: that you have a continuous distribution. It may be any shape at all, and the shape may be unknown.

MR. MILTON L. GODFREY.[3]—I would like to suggest the addition in the list of references of the work done by Mr. E. J. Gumbel at Columbia University.

He has investigated the "law of rare "exceedances,"" as he called it, and he has developed a special probability paper which can be used easily and quickly for plotting actual test points.

The references are:

"Statistical Theory of Extreme Values and Some Practical Applications," *Applied Mathematics Series 33*, Nat. Bureau of Standards, Feb., 12, 1954.

"Probability Tables for the Analysis of Extreme-Value Data," *Applied Mathematics Series 22*, Nat. Bureau of Standards, July 6, 1953.

J. Lieblein, "A New Method of Analyzing Extreme Value Data," *Technical Note No. 3053*, Nat. Advisory Committee Aeronautics (1954).

MR. HERBERT T. CORTEN.[4]—I would like to congratulate the author on a paper that will be valuable to the engineer. While the methods suggested may not satisfy all of the requirements of mathematical statistics, I do not think the engineer will be misled in evaluating materials and making decisions based on small samples of data. The important consideration of the interrelation between the mean value and the scatter in evaluating a material properly is introduced for situations in which the distribution function is not known. It may be that the method can be developed still further; however, in its present form, it constitutes the first step in the direction of providing the engineer with a simple and usable tool.

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[2] Bell Telephone Laboratories, New York, N. Y.

[3] Manager of Development, Talon Inc., Meadville, Pa.

[4] Associate Professor, Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

MR. R. B. MURPHY[5] (*by letter*).—That part of the author's technique which is summarized in Table I may be viewed as a generalization for several samples of a technique presented in 1942 in a now famous paper by S. S. Wilks on the subject of tolerance intervals.[6] Considerable work on this kind of nonparametric technique has been done since. E. J. Gumbel, one of the pioneers in the use of ordered values in statistics, has assembled a pertinent general bibliography,[7] while more recently D. A. S. Fraser has given references in his new book to work on the problem of tolerance intervals.[8]

Miss Torrey's discussion elsewhere reflects justifiable concern with the uses the author has made of his basic material in this paper. I wish to expand on only one comment in this discussion. It is quite possible to doubt the conclusion that there is a significant difference between material B and material C with respect to breaking strength. This doubt is evidenced by a nonparametric test based on the same mathematical assumption as the author's work on "exceedances," or as they are more widely known, coverages.[9] This test is based on a study of the low end of the breaking strength distribution—a region which may reasonably be contended to be of great interest in such a case. The nonparametric test is as follows: plot the sample values from material B and C on the same linear scale, and observe that only one value from material C is less than the least value for material B. Does this indicate that the breaking strengths for material C are shifted upward with respect to those of material B? The probability that if the two sets of breaking strengths came from the same distribution *no* value from the sample of material C would be less than the least of those in material B is approximately 0.21. In this case it would appear unwarranted to draw a conclusion that there is a significant difference between the two materials. There is even less justification for drawing this conclusion when one or more values of breaking strengths from material C is less than the least from material B. If we are forced to choose between materials regardless of this evidence or lack of it, we would make the same choice as in this paper by a simple plot on normal probability paper. The true risks of error for the two more complex methods used by the author to perceive a difference between materials B and C appear to be unknown.

MR. EVAN H. SCHUETTE (*author's closure*).—I offer my sincere thanks to all the discussers for their contributions. I am delighted that people who know something about statistics have taken the time to discuss the matter with those of us whose approaches tend to be strictly of the engineering variety and often not completely rigorous.

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[5] Bell Telephone Laboratories, Inc.,

[6] S. S. Wilks, "Statistical Prediction with Specific Reference to the Problem of Tolerance Limits," *Annals of Mathematical Statistics*, Vol. 13, pp. 400–409.

[7] E. J. Gumbel, "Statistical Theory of Extreme Values and Some Practical Applications," *Applied Mathematics Series No. 33*, Nat. Bureau of Standards (1954).

[8] D. A. S. Fraser, "Non Parametric Methods in Statistics," John Wiley & Sons, Inc., New York, N. Y., p. 159 (1957).

[9] In 1947 J. W. Tukey coined the term "coverage" for what has been called "exceedance" in this paper. The term "exceedance" was used first by Gumbel to denote the number of observations in a sample which exceed a specified ordered value in another sample from the same universe. The standard use of these terms not only in mathematical statistics but in various fields of engineering implies these meanings.

In particular, the additional references that have been offered should be of great value in promoting further mutual understanding. Their omission from my paper should not be taken as a claim to complete originality, but rather constitutes a confession of my total ignorance of the more formal methods and the literature dealing with them.

It is perhaps this very ignorance alone that impels me to the boldness of objecting in one particular. Mr. Murphy implies a preference for "tolerance" and "coverage" where I have used the terms "confidence level" and "exceedance," respectively. He has the backing of established usage for his suggestion.

However, my paper is intended as a simple tool for the research or test engineer who has found neither the time nor the inclination to familiarize himself with methods and terminology of formal statistics. From his point of view, it seems to me the four terms in question might be understood as follows:

Tolerance = permissible variation from a specified nominal value,
Confidence = the degree of assurance that he is right,
Coverage = the completeness with which a given subject has been covered by a given attack, and
Exceedance = the amount exceeding.

If these are reasonable approximations to the general interpretation given to the terms, and if it is one's purpose to provide a method whose use can be readily understood by all, then the terms I have chosen have a distinct advantage. I prefer to leave this question open to be settled by future common usage.

The point was made by several discussors that the paper cites an example wherein one material was shown superior to two others, while statistical methods demonstrate that there are no significant differences among the three. The correctness of their methods I must concede unequivocally. Here, however, as in many other cases I have encountered, a great deal hinges on knowing with precision the question to which we are seeking an answer. The development as given is perhaps remiss in not stating more clearly what question is under consideration.

If the question is: "Are there significant differences demonstrated between any one of these materials and any other?" then the answer, as given by Miss Torrey and Messrs. Shombert and Murphy, must be "No."

If, however, the question is "With which of these materials can I be most confident in saying that at least $x$ per cent of the product will have a breaking strength in excess of $y$ lb?" then the methods of this paper are the straightforward route to the answer.

To state the distinction in another way, the methods of the statistician are completely objective and impersonal. My own methods, as given, are subjective and highly personal. The effect of this subjectiveness is made clear if it is assumed that the part in the example cited is a clevice attachment supporting a man-lift. Let the reader suppose that his weight, plus that of the lift car, is 350 lb. He has to board the car 20 stories up from the bottom of its shaft. Which material will he select for the clevice attachment?

Here, the scarcity of test information on one material and the greater amount on another will definitely influence his judgment. The extent to which this difference should rationally affect the confidence with which he steps aboard the car is indicated by the results that were cited.

# A COOPERATIVE INVESTIGATION OF THE METHOD OF TEST FOR COMPRESSIVE STRENGTH OF BITUMINOUS MIXTURES*

BY CHARLES E. PROUDLEY AND H. FRED WALLER, JR.[1]

## SYNOPSIS

Bituminous paving specifications for North Carolina include a stability requirement based on ASTM Method D 1074 - 55 T.[2] Commercial laboratories representing contractors have found it impossible to obtain stability results that were consistently in agreement with the State Highway Laboratory's values, occasionally varying as much as 50 per cent. The contractual relationships have suffered to some degree, especially since all laboratories claimed to have used the procedures described in Method D 1074.

Laboratories having an interest in bituminous paving work in North Carolina readily agreed to cooperate in an investigation to determine the cause of the discrepancies and to apply remedies if possible. As many as seven laboratories have worked on the problem for more than two years developing data of some significance.

Phases of the cooperative testing include: (1) each laboratory conducting complete tests for density and compressive strength using identical aggregates, asphalt, and mixture formulation; (2) all specimens made in one laboratory and distributed to the cooperating laboratories for density and compression strength determination; (3) specimens made in each of the cooperating laboratories and sent to the North Carolina Laboratory for density and compressive strength.

The data and discussion presented in this paper indicate that Method D 1074 should be further revised to describe details of preparation of the test specimens and their manipulation throughout the test procedure more precisely with a minimum of option for the laboratory technician.

## INVESTIGATION OF AVAILABLE METHODS OF PAVING MIX DESIGN

The years immediately following World War II saw a highway system that was in poor repair and severely overloaded. Pavements that had only a few years earlier been considered adequate for all anticipated traffic were beginning to fail due to the increase in volume of traffic as well as the increase in allowable axle loads. It was readily apparent that highway engineers must make immediate changes in the design concepts if the highway system was to keep pace with traffic demands. More and more the need for a well-organized design procedure, coupled with improved construction practices, was realized.

In North Carolina, as in many other states, it had been a general policy to rely largely on experience in establishing formulas for bituminous paving with little or no regard given to any scientific method of design. While the value of experience should not be minimized, neither should it be used as the sole basis for deciding the various factors involved in selecting job mix formulas. Engineers had apparently failed to recognize the value of the various tests available for measuring certain desirable properties of bituminous mixtures.

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* Presented at the Sixtieth Annual Meeting of the Society, June 16-21, 1957.

[1] State Materials Engineer and Materials Engineer, respectively, State Highway and Public Works Commission, Raleigh, N. C.

[2] Tentative Method of Test for Compressive Strength of Bituminous Mixtures (D 1074 - 55 T), 1955 Book of ASTM Standards, Part 3, p. 1564.

About 1947 the research laboratory of the North Carolina State Highway Commission began studies of several design methods beginning with the 2-in. and 6-in. Hubbard-Field stability test methods and a limited examination of the Marshall stability apparatus. During this period none of the tests were used for predetermining job mix formulas but were intended rather to familiarize the technicians with the techniques and, also, to compile comparative information through which the test values could be interpreted for subsequent preparation of construction specifications. Materials from the various active projects were brought into the laboratory for preparation of test specimens, and the data derived therefrom were studied and efforts made to correlate the information with service performance and properties of the mixture laid in the field as determined from field samples.

Due to poor reproducibility of the 6-in. modified Hubbard-Field method, necessary for bituminous concrete mixtures, this procedure was discontinued after approximately two years. Random particles of coarse aggregate catching on the orifice of the test mold often caused erratic values so that the test could not be reproduced with acceptable accuracy.

At the same time the Marshall test was being used for studying bituminous concrete and sand asphalt mixtures. The authors' chief criticism of this test, insofar as our own laboratory experience is concerned, and one of the reasons for discontinuing the study of its use was the unrealistic (too high) density value produced by the drop hammer as compared with the field compaction of the same mixture in service. The 2-in. Hubbard-Field method appears to provide a relatively dependable measure of some of the desirable properties of fine aggregate mixtures. This method has been used with fairly good success, and during the past five years many sand asphalt projects have been let to contract with a stability requirement based on this procedure. The complete test method is described in the Manual Series No. 2 as published by the Asphalt Institute.[3]

The laboratory, still not being satisfied with procedures investigated for bituminous concrete design, continued its search for a method that would satisfy the need for a procedure which would yield test values that could be reproduced within a workable range of accuracy. Thus a program involving the use of the compression test (ASTM Method D 1074)[2] was instigated. Standard laboratory equipment, except for the mold, may be used for this test, making it relatively inexpensive and the test procedure does not require specialized technical skill.

The test is made on cylindrical specimens 4-in. high and 4-in. in diameter molded under a static load of 3000 psi for 2 min by the double-end plunger method. After an oven curing period of 24 hr at 140 F, the specimens are brought to a temperature of 77 F and tested at a loading rate of 0.2 in. per min until the maximum strength reading is reached. Normally, three specimens are made for each increment of asphalt and a minimum of three increments are used in order to permit drawing a curve. Asphalt is usually increased in increments of $\frac{1}{2}$ per cent, and if necessary interpolations are made for amounts between each ½ per cent.

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[3] Hubbard-Field Method of Mix Design, Mix Design Method of Hot-Mix Asphalt Paving, The Asphalt Institute, p. 39, April, 1956.

## COMPRESSION TEST ADOPTED AS STANDARD

From 1950 through 1954 several hundred bituminous mixtures were made and studied with the aid of the compression test. During this period the approved job mix formulas for state highway projects were not based solely on the test results, although in many cases the test values had a definite bearing on selection of a certain bitumen content or aggregate blend to be used. The technicians became quite proficient in the manipulation of the test details and were able to duplicate results within reasonably narrow limits.

On January 1, 1955, bituminous paving specifications were revised to include a stability requirement based on Method D 1074. Minimum limits were set at 200 psi for binder mixtures and 250 psi for surface mixtures. Where conditions warrant, the stability requirements are increased to a minimum of 250 psi for the binder and 300 psi for the surface. Past experience has shown that more than half the projects awarded since the inclusion of stability requirements have had the higher values, that is, 250 and 300 psi.

## COOPERATIVE TESTS PLANNED

Shortly after a stability requirement based on ASTM Method D 1074 became a part of the North Carolina standard specifications for bituminous paving, discrepancies began appearing between results obtained by commercial laboratories and those obtained by the Highway laboratory. Usually results obtained by the commercial laboratories were higher, often by as much as 25 to 50 per cent. Since the paving contractors use the values furnished by the commercial laboratories for bidding purposes it is imperative that they be furnished results that are reasonably reliable. There have been several occasions where a contractor was the successful bidder on a project, his bid being based on results furnished by a commercial laboratory. When his proposed materials were submitted to the Highway laboratory for test they would occasionally be rejected for failure to meet the minimum stability requirements. This not only caused strained relations between the contractor and the two laboratories involved but it instilled a lack of confidence in the contractor's mind as to the reliability of the test method. The Highway Department was criticized for adopting a method about which so little was known. It was our feeling that as the other laboratories gained experience in performing the test, the variations would diminish and perhaps disappear. In the meantime, however, the contractors stood the possibility of losing money by being required to add more expensive materials than they had originally anticipated.

We believed the best approach to the problem would be a "round robin" series with each of the interested laboratories participating. In the beginning, four laboratories, including our own, were involved, and as the tests progressed three others joined the group, making a total of seven.

As a matter of interest the 2-in. Hubbard-Field test was included in the cooperative study. These results will not be discussed at this time but are available for those who are interested in reviewing these data.

The entire investigation was based on typical North Carolina aggregates with no unusual or tricky mixes involved. A wide variety of materials was used so that both good and questionable aggregates could be studied to see if there was any particular aggregate characteristic

TABLE I.—ASTM D 1074 - 55 T[2] COMPRESSION TEST, PHASE I.

All specimens molded and tested by indicated laboratories using aggregates and formulations furnished by North Carolina Laboratory. Job mix formulas for each series are shown in Table II. All stability values shown in psi.

| Laboratory | Series 1 | | Series 2 | | Series 3 | | Series 4 | | Series 5 | | Series 6 | | Series 7 | | Series 8 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Specific Gravity | Stability | Specific Gravity | Stability | Specific Gravity | Stability | Specific Gravity | Stability | Specific Gravity | Stability | Specific Gravity | Stability | Specific Gravity | Stability | Specific Gravity | Stability |
| N. C. Highway Department | 2.315 | 252 | 2.333 | 369 | 2.365 | 303 | 2.205 | 127 | 2.176 | 352 | 2.277 | 320 | 2.299 | 311 | 2.284 | 209 |
| | 2.314 | 247 | 2.340 | 360 | 2.377 | 315 | 2.195 | 124 | 2.187 | 342 | 2.274 | 311 | 2.203 | 311 | 2.286 | 218 |
| | 2.314 | 245 | 2.340 | 373 | 2.378 | 334 | 2.195 | 130 | 2.186 | 352 | 2.272 | 325 | 2.301 | 296 | 2.280 | 210 |
| | | | | | 2.366 | 311 | | | | | | | | | | |
| | | | | | 2.373 | 315 | | | | | | | | | | |
| | | | | | 2.364 | 325 | | | | | | | | | | |
| Average | 2.314 | 248 | 2.338 | 367 | 2.371 | 317 | 2.198 | 127 | 2.183 | 349 | 2.274 | 319 | 2.298 | 306 | 2.283 | 212 |
| A | 2.317 | 202 | 2.347 | 317 | 2.379 | 298 | 2.214 | 154 | 2.205 | 413 | 2.294 | 376 | 2.307 | 368 | 2.320 | 239 |
| | 2.313 | 203 | 2.353 | 357 | 2.382 | 290 | 2.214 | 167 | 2.204 | 399 | 2.292 | 372 | 2.301 | 362 | 2.307 | 239 |
| | 2.304 | 210 | | | 2.372 | 305 | 2.216 | 180 | 2.216 | 415 | 2.291 | 365 | | | | |
| | | | | | | | 2.207 | 179 | | | | | | | | |
| Average | 2.311 | 205 | 2.350 | 337 | 2.377 | 298 | 2.213 | 170 | 2.208 | 409 | 2.292 | 371 | 2.304 | 365 | 2.314 | 239 |
| B | 2.298 | 219 | 2.32 | 326 | 2.331 | 316 | 2.196 | 141 | 2.183 | 375 | 2.265 | 314 | 2.293 | 309 | 2.265 | 220 |
| | 2.295 | 221 | 2.33 | 340 | 2.332 | 314 | 2.194 | 143 | 2.178 | 378 | 2.275 | 321 | 2.295 | 312 | 2.261 | 235 |
| | 2.300 | 229 | 2.33 | 334 | 2.340 | 320 | 2.198 | 149 | 2.174 | 370 | 2.272 | 312 | 2.297 | 321 | 2.268 | 260 |
| Average | 2.298 | 223 | 2.33 | 333 | 2.334 | 317 | 2.196 | 144 | 2.178 | 374 | 2.271 | 316 | 2.295 | 314 | 2.265 | 238 |
| C | 2.30 | 240 | 2.34 | 350 | 2.34 | 348 | 2.20 | 190 | 2.220 | 341 | 2.282 | 282 | 2.300 | 319 | 2.301 | 281 |
| | 2.31 | 248 | 2.33 | 340 | 2.34 | 345 | 2.20 | 186 | 2.199 | 325 | 2.277 | 273 | 2.298 | 310 | 2.302 | 252 |
| | 2.30 | 242 | 2.33 | 342 | 2.35 | 352 | 2.21 | 191 | 2.195 | 333 | 2.280 | 276 | 2.301 | 325 | 2.299 | 255 |
| | | | 2.33 | 353 | | | | | | | | | | | | |
| | | | 2.34 | 349 | | | | | | | | | | | | |
| Average | 2.30 | 243 | 2.33 | 347 | 2.34 | 348 | 2.20 | 189 | 2.204 | 333 | 2.280 | 277 | 2.300 | 318 | 2.301 | 263 |
| D | | | | | | | | | 2.204 | 344 | 2.29 | 366 | | | 2.38 | 281 |
| | | | | | | | | | 2.209 | 350 | 2.31 | 366 | | | 2.36 | 255 |
| | | | | | | | | | 2.215 | 394 | 2.31 | 366 | | | 2.35 | 245 |
| Average | | | | | | | | | 2.209 | 363 | 2.30 | 366 | | | 2.36 | 260 |
| E | | | | | | | | | 2.184 | 291 | 2.287 | 281 | 2.298 | 273 | 2.260 | 202 |
| | | | | | | | | | 2.189 | 322 | 2.283 | 274 | 2.301 | 269 | 2.274 | 222 |
| | | | | | | | | | 2.188 | 307 | | | | | | |
| | | | | | | | | | 2.178 | 313 | 2.280 | 281 | 2.300 | 274 | 2.272 | 202 |
| Average | | | | | | | | | 2.185 | 308 | 2.283 | 279 | 2.300 | 272 | 2.269 | 209 |
| Over-all Average | | 230 | | 346 | | 320 | | 158 | | 356 | | 321 | | 315 | | 237 |
| High Value | | 248 | | 367 | | 348 | | 189 | | 409 | | 371 | | 365 | | 263 |
| Low Value | | 205 | | 333 | | 298 | | 127 | | 308 | | 277 | | 272 | | 209 |
| Maximum Variation from Average per cent | | 10 | | 6 | | 9 | | 20 | | 15 | | 16 | | 16 | | 12 |

that could account for the excessive variations. The materials and combinations as used would meet specification requirements for Los Angeles abrasion loss, grading, and in most cases, stability.

## PROCEDURES FOR COOPERATIVE TESTS

The first part of the cooperative tests has been called "phase I." This phase covered a period of about eight months. Identical aggregates, along with 85-100 penetration asphalt, were sent to all participants each month with a formula to be used in preparing the test specimens. In this way everyone involved used the same materials, the same formula, and, supposedly, the same procedure for each series. Table I shows the results, both specific gravity and stability, of all test specimens in the first phase while Table II indicates the eight job mix formulas that were used.

TABLE II.—JOB MIX FORMULAS FOR PHASE I.

| | | Series 1[a] | Series 2[b] | Series 3[c] | Series 4[d] | Series 5[e] | Series 6[f] | Series 7[g] | Series 8[h] |
|---|---|---|---|---|---|---|---|---|---|
| Passing: | Retained, per cent: | | | | | | | | |
| $\frac{3}{4}$ in....... | $\frac{1}{2}$ in....... | 2.0 | 0.0 | 0.0 | 18.6 | 2.6 | 2.0 | 2.0 | 18.0 |
| $\frac{1}{2}$ in....... | $\frac{3}{8}$ in....... | 20.0 | 20.4 | 20.4 | 17.4 | 12.4 | 18.0 | 18.0 | 16.8 |
| $\frac{3}{8}$ in....... | No. 4...... | 18.0 | 17.4 | 17.4 | 15.5 | 23.8 | 18.0 | 18.0 | 15.0 |
| No. 4...... | No. 10..... | 14.1 | 13.2 | 13.2 | 10.6 | 13.2 | 15.0 | 15.0 | 10.2 |
| No. 10..... | No. 40..... | 15.6 | 17.9 | 17.9 | 8.2 | 16.5 | 22.1 | 22.1 | 14.3 |
| No. 40..... | No. 80..... | 14.8 | 16.6 | 16.6 | 18.3 | 14.5 | 9.2 | 9.2 | 12.3 |
| No. 80..... | No. 200.... | 6.0 | 4.2 | 4.2 | 6.2 | 6.7 | 4.7 | 4.7 | 5.9 |
| No. 200.... | .... | 4.5 | 4.8 | 4.8 | 0.7 | 4.8 | 5.5 | 5.5 | 2.5 |
| Bitumen, per cent....... | | 5.0 | 5.5 | 5.5 | 4.5 | 5.5 | 5.5 | 5.5 | 5.0 |
| | | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |

[a] Series 1.—Siliceous gravel (Senter) coarse aggregate and local fine aggregate.

[b] Series 2.—Granite coarse aggregate (McLeansville) and local fine aggregate.

[c] Series 3.—Same as series 2.

[d] Series 4.—Siliceous gravel coarse aggregate and commercial fine aggregate (Lilesville).

[e] Series 5.—Marl coarse aggregate that has a considerable amount of the lime replaced with silica. Fine aggregate, screenings, from same source (Belgrade).

[f] Series 6.—Granite coarse aggregate (Rolesville) and granite screenings (Neverson).

[g] Series 7.—Same as series 6.

[h] Series 8.—Granite coarse aggregate (Neverson) and local fine aggregate (Shaw Pit).

Phase II was planned in such manner that it would be easier to detect variations in the major items concerned with performance of the tests. In the first round of phase II, thirty identical specimens were molded and cured by the North Carolina laboratory and five were sent to each of the other laboratories. In order to ensure a minimum of disturbance, the specimens were wrapped in heavy paper and packed in sawdust for shipment. The receiving laboratories needed only to bring the specimens to the standard 77 F test temperature and apply the load at 0.2 in. per min until the maximum load was reached. Theoretically all specimens were identical, and the possible sources of variation would be in the test temperature and the rate of loading.

Results of this series are shown in Table III.

In the second series of phase II, an identical procedure was followed using a different job mix formula. Results of these tests are shown in Table IV.

The third and final series of phase II was divided into three parts. In part one

## TABLE III.—EFFECT OF TEST PROCEDURES AMONG LABORATORIES ON COMPRESSIVE STRENGTH, PHASE II—SERIES 1.

All specimens molded and cured by North Carolina Laboratory. The specimen numbers denote order of preparation starting with one. Specimens were tested by all laboratories after approximately one week's fabrication. The assignment of the individual laboratories was to bring specimens to test temperature (77 F), determine specific gravity and compressive strength. Job mix formula for this series is shown in Table VI.

| | Specimen | Specific Gravity | Stability, psi |
|---|---|---|---|
| N.C. Highway Department | No. 21 | 2.235 | 250 |
| | No. 27 | 2.263 | 299 |
| | No. 3 | 2.237 | 272 |
| | No. 9 | 2.228 | 252 |
| | No. 15 | 2.235 | 261 |
| | Average | 2.240 | 267 |
| A | No. 16 | 2.257 | 288 |
| | No. 22 | 2.254 | 290 |
| | No. 28 | 2.249 | 280 |
| | No. 4 | 2.252 | 290 |
| | No. 10 | 2.254 | 300 |
| | Average | 2.253 | 290 |
| B | No. 6 | 2.244 | 277 |
| | No. 12 | 2.250 | 296 |
| | No. 18 | 2.250 | 278 |
| | No. 24 | 2.251 | 275 |
| | No. 30 | 2.246 | 280 |
| | Average | 2.248 | 281 |
| C | No. 1 | 2.236 | 288 |
| | No. 7 | 2.240 | 307 |
| | No. 13 | 2.241 | 283 |
| | No. 19 | 2.234 | 271 |
| | No. 25 | 2.234 | 244 |
| | Average | 2.237 | 279 |
| D | No. 26 | 2.27 | 251 |
| | No. 2 | 2.26 | 264 |
| | No. 8 | 2.26 | 264 |
| | No. 14 | 2.26 | 259 |
| | No. 20 | 2.26 | 247 |
| | Average | 2.26 | 257 |
| E | No. 11 | 2.244 | 257 |
| | No. 17 | 2.245 | 260 |
| | No. 23 | 2.243 | 255 |
| | No. 29 | 2.245 | 246 |
| | No. 5 | 2.243 | 240 |
| | Average | 2.244 | 252 |

## TABLE IV.—EFFECT OF TEST PROCEDURES AMONG LABORATORIES ON COMPRESSIVE STRENGTH, PHASE II—SERIES 2.

All specimens molded and cured by North Carolina Laboratory. The specimen numbers denote order of preparation starting with one. Specimens were tested by all laboratories after approximately one week's fabrication. The assignment of the individual laboratories was to bring specimens to test temperature (77 F), determine specific gravity and compressive strength. Job mix formula for this series is shown in Table VI.

| | Specimen | Specific Gravity | Stability, psi |
|---|---|---|---|
| N.C. Highway Department | No. 1 | 2.228 | 283 |
| | No. 7 | 2.229 | 274 |
| | No. 13 | 2.226 | 288 |
| | No. 19 | 2.229 | 275 |
| | No. 21 | 2.304 | 298 |
| | Average | 2.243 | 284 |
| A | No. 6 | 2.243 | 324 |
| | No. 12 | 2.234 | 319 |
| | No. 18 | 2.237 | 317 |
| | No. 24 | 2.233 | 326 |
| | No. 30 | 2.240 | 317 |
| | Average | 2.237 | 321 |
| B | No. 11 | 2.234 | 293 |
| | No. 17 | 2.231 | 272 |
| | No. 23 | 2.242 | 303 |
| | No. 29 | 2.246 | 313 |
| | No. 35 | 2.241 | 305 |
| | Average | 2.239 | 297 |
| C | No. 5 | 2.231 | 329 |
| | No. 16 | 2.237 | 317 |
| | No. 22 | 2.235 | 311 |
| | No. 28[a] | 2.243[a] | 220[a] |
| | No. 34 | 2.229 | 302 |
| | Average | 2.233 | 315 |
| D | No. 2 | 2.23 | 288 |
| | No. 8 | 2.23 | 292 |
| | No. 14 | 2.25 | 300 |
| | No. 20 | 2.25 | 296 |
| | No. 31 | 2.25 | 302 |
| | Average | 2.24 | 296 |
| E | No. 3 | 2.234 | 299 |
| | No. 9 | 2.238 | 299 |
| | No. 15 | 2.242 | 304 |
| | No. 26 | 2.244 | 292 |
| | No. 32 | 2.239 | 310 |
| | Average | 2.239 | 301 |
| F | No. 4 | 2.247 | 300 |
| | No. 10 | 2.241 | 300 |
| | No. 25 | 2.237 | 284 |
| | No. 27 | 2.243 | 289 |
| | No. 33 | 2.244 | 298 |
| | Average | 2.242 | 294 |

[a] Not included in average.

## TABLE V.—EFFECT OF TEST PROCEDURES AMONG LABORATORIES ON COMPRESSIVE STRENGTH:

### (*a*) Phase II, Series 3, Part 1

All specimens molded and cured by North Carolina Laboratory. The specimen numbers denote order of preparation starting with one. Specimens were tested by all laboratories after approximately one week's fabrication. The assignment of the individual laboratories was to bring specimens to test temperature (77 F), determine specific gravity and compressive strength. Job mix formula for this series is shown in Table VI.

| | Specimen | Specific Gravity | Stability, psi |
|---|---|---|---|
| N.C. Highway Department | No. 4 | 2.277 | 244 |
| | No. 7 | 2.277 | 242 |
| | No. 13 | 2.292 | 274 |
| | No. 19 | 2.273 | 248 |
| | No. 27 | 2.284 | 260 |
| | Average | 2.281 | 254 |
| A | No. 6 | 2.300 | 273 |
| | No. 12 | 2.290 | 259 |
| | No. 18 | 2.289 | 252 |
| | No. 24 | 2.281 | 248 |
| | No. 30 | 2.300 | 267 |
| | Average | 2.292 | 260 |
| B | No. 11 | 2.275 | 237 |
| | No. 17 | 2.278 | 245 |
| | No. 23 | 2.289 | 269 |
| | No. 29 | 2.278 | 260 |
| | No. 35 | 2.277 | 277 |
| | Average | 2.279 | 258 |
| C | No. 5 | 2.289 | 234 |
| | No. 16 | 2.272 | 217 |
| | No. 22 | 2.277 | 219 |
| | No. 28 | 2.273 | 201 |
| | No. 34 | 2.283 | 241 |
| | Average | 2.279 | 222 |
| D | No. 2 | 2.27 | 328 |
| | No. 8 | 2.28 | 328 |
| | No. 14 | 2.28 | 326 |
| | No. 20 | 2.28 | 305 |
| | No. 31 | 2.27 | 310 |
| | Average | 2.28 | 319 |
| E | No. 3 | 2.288 | 238 |
| | No. 6 | 2.277 | 230 |
| | No. 15 | 2.281 | 238 |
| | No. 26 | 2.282 | 241 |
| | No. 32 | 2.292 | 242 |
| | Average | 2.284 | 238 |
| F | No. 4 | 2.280 | 232 |
| | No. 10 | 2.288 | 226 |
| | No. 25 | 2.278 | 229 |
| | No. 27 | 2.281 | 242 |
| | No. 33 | 2.279 | 228 |
| | Average | 2.281 | 231 |

### (*b*) Phase II, Series 3, Part 2

All specimens molded and tested by indicated laboratories using aggregates and formulations furnished by North Carolina Laboratory. This group of specimens is theoretically identical in proportioning of materials to the two other groups of specimens in Phase II—Series 3. (See Tables V(*a*) and V(*c*).)

| | Specimen | Specific Gravity | Stability, psi |
|---|---|---|---|
| A | No. 2 | 2.300 | 269 |
| | No. 4 | 2.299 | 261 |
| | No. 6 | 2.295 | 250 |
| | No. 8 | 2.299 | 263 |
| | No. 10 | 2.295 | 252 |
| | Average | 2.297 | 259 |
| B | No. 6 | 2.273 | 201 |
| | No. 7 | 2.272 | 183 |
| | No. 8 | 2.275 | 216 |
| | No. 9 | 2.272 | 189 |
| | No. 10 | 2.281 | 203 |
| | Average | 2.275 | 198 |
| C | No. 1 | 2.268 | 240 |
| | No. 2 | 2.277 | 250 |
| | No. 3 | 2.266 | 239 |
| | No. 4 | 2.274 | 237 |
| | No. 5 | 2.276 | 251 |
| | Average | 2.272 | 243 |
| D | No. 1 | 2.33 | 396 |
| | No. 3 | 2.35 | 368 |
| | No. 5 | 2.36 | 378 |
| | No. 7 | 2.36 | 360 |
| | No. 9 | 2.30 | 374 |
| | Average | 2.34 | 375 |
| E | No. 1 | 2.300 | 230 |
| | No. 3 | 2.294 | 236 |
| | No. 5 | 2.300 | 237 |
| | No. 7 | 2.295 | 241 |
| | No. 9 | 2.292 | 227 |
| | Average | 2.296 | 234 |
| F | No. 3 | 2.277 | 250 |
| | No. 5 | 2.271 | 256 |
| | No. 6 | 2.272 | 258 |
| | No. 8 | 2.263 | 241 |
| | No. 10 | 2.273 | 247 |
| | Average | 2.271 | 250 |

**(Table V Continued on p. 1328)**

TABLE V.—*Concluded.*

(*c*) PHASE II, SERIES 3, PART 3.

All specimens in this group were molded by indicated laboratories using aggregates and formulations furnished by the North Carolina Laboratory. The specimens were tested, however, by the North Carolina Laboratory. This group of specimens is theoretically identical in proportioning of materials to the other two groups in Phase II—Series 3 (see Table V(*a*) and V(*b*).)

| | Specimen | Specific Gravity | Stability, psi |
|---|---|---|---|
| A | No. 1 | 2.285 | 245 |
| | No. 3 | 2.296 | 240 |
| | No. 5 | 2.291 | 255 |
| | No. 7 | 2.287 | 247 |
| | No. 9 | 2.287 | 252 |
| | Average | 2.289 | 248 |
| B | No. 1 | 2.269 | 232 |
| | No. 2 | 2.260 | 229 |
| | No. 3 | 2.257 | 225 |
| | No. 4 | 2.263 | 229 |
| | No. 5 | 2.269 | 245 |
| | Average | 2.264 | 232 |
| C | No. 6 | 2.273 | 225 |
| | No. 7 | 2.311 | 269 |
| | No. 8 | 2.284 | 258 |
| | No. 9 | 2.322 | 272 |
| | No. 10 | 2.288 | 283 |
| | Average | 2.296 | 261 |
| D | No. 2 | 2.318 | 314 |
| | No. 4 | 2.322 | 314 |
| | No. 6 | 2.323 | 340 |
| | No. 8 | 2.320 | 299 |
| | No. 10 | 2.323 | 299 |
| | Average | 2.321 | 313 |
| E | No. 2 | 2.290 | 231 |
| | No. 4 | 2.277 | 233 |
| | No. 6 | 2.276 | 231 |
| | No. 8 | 2.274 | 231 |
| | No. 10 | 2.279 | 234 |
| | Average | 2.279 | 232 |
| F | No. 1 | 2.265 | 244 |
| | No. 2 | 2.271 | 252 |
| | No. 4 | 2.273 | 258 |
| | No. 7 | 2.274 | 266 |
| | No. 9 | 2.278 | 261 |
| | Average | 2.272 | 256 |

all specimens were molded and cured by the North Carolina Highway laboratory and five each sent to the other laboratories for testing. In part two, each laboratory made and tested specimens using the same formula and identical aggregates and asphalt as was used in part one; in part three each participating laboratory made and cured five specimens with identical materials and sent them to the North Carolina Highway laboratory for testing. Theoretically, all specimens in each of the three groups were identical regardless of who molded or tested them.

TABLE VI.—JOB MIX FORMULAS FOR PHASE II.

| | | Series 1[a] | Series 2[b] | Series 3[c] |
|---|---|---|---|---|
| Passing: | Retained, per cent: | | | |
| 3/4 in. | 1/2 in. | 2.4 | 4.8 | 6.3 |
| 1/2 in. | 3/8 in. | 9.6 | 14.3 | 7.8 |
| 3/8 in. | No. 4 | 26.3 | 19.0 | 27.7 |
| No. 4 | No. 10 | 15.0 | 14.3 | 10.5 |
| No. 10 | No. 40 | 19.3 | 23.2 | 12.9 |
| No. 40 | No. 80 | 10.3 | 9.9 | 18.6 |
| No. 80 | No. 200 | 7.3 | 6.2 | 5.8 |
| No. 200 | .... | 5.3 | 3.3 | 5.4 |
| Bitumin, per cent | .... | 4.5 | 5.0 | 5.0 |
| | .... | 100.0 | 100.0 | 100.0 |

[a] Series 1.—Granite coarse and fine aggregate (Rolesville stone and screenings).

[b] Series 2.—Granite coarse and fine aggregate (Neverson stone and screenings).

[c] Series 3.—Siliceous gravel coarse aggregate (Marlboro) and local fine aggregate.

Table VI shows the job mix formula used for each of the three series or rounds of phase II while Tables V (*a*), (*b*), and (*c*) show the individual test results.

## DISCUSSION OF TEST DATA

For the first three months the variations among laboratories in phase I appeared reasonable, that is, they were no greater than the repeatability of the test itself. Our own laboratory has developed a degree of repeatability of not more than 10 per cent and, in most cases, in the range of 5 per cent. During the first three series of phase I, the maximum variation from the average stability proved to be 10, 6, and 9 per cent respectively. In series 4 of this phase the maximum variation from the average went up to 20 per cent. A spread of values this great would render the test questionable in attempting to accept or reject a specific mixture on the basis of stability alone, particularly when a borderline mixture is involved. Beginning with series 5, two additional laboratories joined the group. The maximum variation for series 5, 6, 7, and 8, considering only the original four laboratories, was 12, 16, 12, and 11 per cent respectively. When all six laboratories are considered these variations advance to 15, 16, 16, and 15 per cent respectively. In practically all cases the laboratories were able

TABLE VII.—SUMMARY OF COMPRESSION TEST RESULTS, PHASE I.

Data from Table I.

| Laboratory | Average Stability psi | Deviation, per cent | | Average Stability, psi | Deviation, per cent | | Average Stability, psi | Deviation, per cent | | Average Stability, psi | Deviation, per cent | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | *a* | *b* | | *a* | *b* | | *a* | *b* | | *a* | *b* |
| | SERIES 1 | | | SERIES 2 | | | SERIES 3 | | | SERIES 4 | | |
| N.C. | 248 | 7.83 | 1.07 | 367 | 6.07 | 1.36 | 317 | 0.94 | 2.58 | 127 | 19.62 | 1.57 |
| A | 205 | 10.87 | 1.63 | 337 | 2.60 | 5.93 | 298 | 6.88 | 1.68 | 170 | 7.59 | 5.58 |
| B | 223 | 3.04 | 1.79 | 333 | 3.76 | 1.50 | 317 | 0.94 | 0.74 | 144 | 8.86 | 2.08 |
| C | 243 | 5.65 | 1.23 | 347 | 0.29 | 1.33 | 348 | 8.75 | 0.67 | 189 | 19.62 | 1.06 |
| Average | 230 | 6.85 | 1.43 | 346 | 3.18 | 2.53 | 320 | 4.38 | 1.42 | 158 | 13.92 | 2.57 |
| Maximum | 248 | 10.87 | 1.79 | 367 | 6.07 | 5.93 | 348 | 8.75 | 2.58 | 189 | 19.62 | 5.58 |
| Minimum | 205 | 3.04 | 1.07 | 333 | 0.29 | 1.33 | 298 | 0.94 | 0.67 | 127 | 7.59 | 1.06 |
| Range | 43 | 7.83 | 0.72 | 34 | 5.78 | 4.60 | 50 | 7.81 | 1.91 | 62 | 12.03 | 4.52 |
| Estimated standard deviation (*s*) | 19.72 | .... | .... | 15.18 | .... | .... | 20.70 | .... | .... | 27.45 | .... | .... |
| Coefficient of variation, per cent | 8.57 | .... | .... | 4.39 | .... | .... | 6.47 | .... | .... | 17.37 | .... | .... |
| | SERIES 5 | | | SERIES 6 | | | SERIES 7 | | | SERIES 8 | | |
| N.C. | 349 | 1.97 | 1.24 | 319 | 0.62 | 1.57 | 306 | 2.86 | 2.18 | 212 | 10.55 | 1.73 |
| A | 409 | 14.89 | 1.63 | 371 | 15.58 | 1.08 | 365 | 15.87 | 0.82 | 239 | 0.84 | 0.0 |
| B | 374 | 5.06 | 0.80 | 316 | 1.56 | 1.16 | 314 | 0.32 | 1.49 | 238 | 0.42 | 6.02 |
| C | 333 | 6.46 | 1.60 | 277 | 13.71 | 1.20 | 318 | 0.95 | 1.67 | 263 | 10.97 | 4.69 |
| D | 363 | 1.97 | 5.78 | 366 | 14.02 | 0.0 | .... | .... | .... | 260 | 9.70 | 5.26 |
| E | 308 | 13.48 | 3.00 | 279 | 13.08 | 1.08 | 272 | 13.65 | 0.74 | 209 | 11.81 | 4.31 |
| Average | 356 | 7.30 | 2.34 | 321 | 9.76 | 1.01 | 315 | 6.73 | 1.38 | 237 | 7.38 | 3.67 |
| Maximum | 409 | 14.89 | 5.78 | 371 | 15.58 | 1.57 | 365 | 15.87 | 2.18 | 263 | 11.81 | 6.02 |
| Minimum | 308 | 1.97 | 0.80 | 277 | 0.62 | 0.0 | 272 | 0.32 | 0.74 | 209 | 0.84 | 0.0 |
| Range | 101 | 12.92 | 4.98 | 94 | 14.96 | 1.57 | 93 | 15.55 | 1.44 | 54 | 10.97 | 6.02 |
| Estimated standard deviation, *s* | 34.82 | .... | .... | 40.63 | .... | .... | 33.31 | .... | .... | 22.86 | .... | .... |
| Coefficient of variation, per cent | 9.78 | .... | .... | 12.66 | .... | .... | 10.57 | .... | .... | 9.65 | .... | .... |

[a] Percentage variation among cooperating laboratories.

[b] Percentage variation for the individual laboratory results.

TABLE VIII.—REPEATABILITY—PHASE II STABILITY DATA.

This table indicates the per cent variation within each group of five specimens.

| Laboratories | Specimen No. 1 | Specimen No. 2 | Specimen No. 3 | Specimen No. 4 | Specimen No. 5 | Average | $P$ = 0.95 Confidence Limits | Range | Standard Deviation | Coefficient of Variation, per cent |
|---|---|---|---|---|---|---|---|---|---|---|
| PHASE II, SERIES 1. (All specimens molded by N. C. Laboratory.) | | | | | | | | | | |
| N.C. Highway Department | 250 | 299 | 272 | 252 | 261 | 266.8 | 294.4<br>239.2 | 49 | 20.0 | 7.50 |
| A | 288 | 290 | 280 | 290 | 300 | 289.6 | 299.4<br>279.8 | 20 | 7.10 | 2.45 |
| B | 277 | 296 | 278 | 275 | 280 | 281.2 | 292.8<br>269.6 | 21 | 8.41 | 2.99 |
| C | 288 | 307 | 283 | 271 | 244 | 278.6 | 310.8<br>246.4 | 63 | 23.30 | 8.36 |
| D | 251 | 264 | 264 | 259 | 247 | 257.0 | 267.6<br>246.4 | 17 | 7.70 | 3.00 |
| E | 257 | 260 | 255 | 246 | 240 | 251.6 | 263.1<br>240.1 | 20 | 8.32 | 3.31 |
| PHASE II, SERIES 2. (All specimens molded by N.C. Laboratory.) | | | | | | | | | | |
| N.C. Highway Department | 283 | 274 | 288 | 275 | 298 | 283.6 | 297.1<br>270.1 | 24 | 9.8 | 3.45 |
| A | 324 | 319 | 317 | 326 | 317 | 320.6 | 326.3<br>314.9 | 9 | 4.15 | 1.29 |
| B | 293 | 272 | 303 | 313 | 305 | 297.2 | 319.0<br>275.4 | 41 | 15.8 | 5.32 |
| C | 329 | 317 | 311 | 220 | 302 | 295.8 | 355.8<br>235.8 | 107 | 43.5 | 14.71 |
| D | 288 | 292 | 300 | 296 | 302 | 295.6 | 303.5<br>287.7 | 14 | 5.7 | 1.93 |
| E | 299 | 299 | 304 | 292 | 310 | 300.8 | 310.0<br>291.6 | 18 | 6.69 | 2.22 |
| F | 300 | 300 | 284 | 289 | 298 | 294.2 | 304.3<br>284.1 | 16 | 7.3 | 2.48 |

Table VIII.—*continued*

| Laboratories | Specimen No. 1 | Specimen No. 2 | Specimen No. 3 | Specimen No. 4 | Specimen No. 5 | Average | P = 0.95 Confidence Limits | Range | Standard Deviation | Coefficient of Variation, per cent |
|---|---|---|---|---|---|---|---|---|---|---|
| Phase II, Series 3, Part 1. (All specimens molded by N.C. Laboratory.) | | | | | | | | | | |
| N.C. Highway Department | 244 | 242 | 274 | 248 | 260 | 253.6 | 272.0<br>235.2 | 32 | 13.3 | 5.24 |
| A | 273 | 259 | 252 | 248 | 267 | 259.8 | 274.0<br>215.6 | 25 | 10.3 | 3.96 |
| B | 237 | 245 | 269 | 260 | 277 | 257.6 | 280.4<br>234.8 | 40 | 16.5 | 6.40 |
| C | 234 | 217 | 219 | 201 | 241 | 222.4 | 243.9<br>200.9 | 40 | 15.6 | 7.01 |
| D | 328 | 328 | 326 | 305 | 310 | 319.4 | 334.6<br>304.2 | 23 | 11.0 | 3.44 |
| E | 238 | 230 | 238 | 241 | 242 | 237.8 | 244.3<br>231.3 | 12 | 4.71 | 1.98 |
| F | 232 | 226 | 229 | 242 | 228 | 231.4 | 240.1<br>222.7 | 16 | 6.3 | 2.72 |
| Phase II, Series 3, Part 2. (All specimens molded and tested by indicated Laboratory.) | | | | | | | | | | |
| A | 269 | 261 | 250 | 263 | 252 | 259.0 | 269.9<br>248.1 | 19 | 7.9 | 3.05 |
| B | 201 | 183 | 216 | 189 | 203 | 198.4 | 216.1<br>180.7 | 33 | 12.8 | 6.45 |
| C | 240 | 250 | 239 | 237 | 251 | 243.4 | 252.4<br>234.4 | 14 | 6.57 | 2.70 |
| D | 396 | 368 | 378 | 360 | 374 | 375.2 | 393.8<br>356.6 | 36 | 13.46 | 3.59 |
| E | 230 | 236 | 237 | 241 | 227 | 234.2 | 242.0<br>226.4 | 14 | 5.63 | 2.40 |
| F | 250 | 256 | 258 | 241 | 247 | 250.4 | 259.9<br>240.9 | 17 | 6.9 | 2.75 |
| Phase II, Series 3, Part 3. (Specimens molded by indicated Laboratories and Tested by N.C. Laboratory.) | | | | | | | | | | |
| A | 245 | 240 | 255 | 247 | 252 | 247.8 | 255.9<br>239.7 | 15 | 5.9 | 2.38 |
| B | 232 | 229 | 225 | 229 | 245 | 232.0 | 242.6<br>211.4 | 20 | 7.7 | 3.32 |
| C | 225 | 269 | 258 | 272 | 283 | 261.4 | 292.0<br>230.8 | 58 | 22.2 | 8.49 |
| D | 314 | 314 | 340 | 299 | 299 | 313.2 | 336.3<br>290.1 | 41 | 16.75 | 5.35 |
| E | 231 | 233 | 231 | 231 | 234 | 232.0 | 233.9<br>230.1 | 3 | 1.41 | 0.61 |
| F | 244 | 252 | 258 | 266 | 261 | 256.2 | 267.9<br>244.5 | 22 | 8.5 | 3.32 |

to check the specific gravity of the compacted specimens within satisfactorily narrow limits.

Table VII presents a summary of compression test results for phase I. This table shows the average per cent deviation, first between laboratories and second between individual laboratory results.

The statistician who made this analysis called attention to the fact that the use of only three specimens was not adequate to furnish information for a reliable statistical interpretation. Therefore, future tests were planned based on a minimum of five specimens.

TABLE IX.—REPRODUCIBILITY—PHASE II STABILITY DATA.
This table indicates the per cent variation between individual laboratories.

| Series | N.C. Lab. | Lab. A | Lab. B | Lab. C | Lab. D | Lab. E | Lab. F. | Average | $P = 0.95$ Confidence Limits | Range | Standard Deviation | Coefficient of Variation, Per cent |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1............ | 266.8 | 289.6 | 281.2 | 278.6 | 257.0 | 251.6 | ... | 270.8 | 287.8<br>253.8 | 38.0 | 14.8 | 5.47 |
| 2............ | 283.6 | 320.6 | 297.2 | 295.8 | 295.6 | 300.8 | 294.2 | 298.26 | 209.46<br>278.06 | 37.0 | 11.2 | 3.76 |
| 3, Part 1...... | 253.6 | 259.8 | 257.6 | 222.4 | 319.4 | 237.8 | 231.4 | 254.6 | 286.4<br>222.8 | 97.0 | 31.8 | 12.49 |
| 3, Part 2...... | ... | 259 | 198.4 | 243.4 | 375.2 | 234.2 | 250.4 | 260.1 | 329.1<br>191.1 | 176.8 | 60.0 | 23.07 |
| 3, Part 3...... | ... | 247.8 | 232 | 261.4 | 313.2 | 232.0 | 256.2 | 257.1 | 291.6<br>222.6 | 81.2 | 30.0 | 11.67 |

Note that phase I was concerned entirely with specimens that were molded and tested by each participating laboratory using aggregates and asphalt furnished by the North Carolina laboratory and the results from all laboratories should have been the same.

In considering the stability values derived from phase II, it appears that the per cent deviation from the average value in each group lies within the range of 6 to 8 per cent when all the specimens are made by one organization and tested by others. Obviously the variations increase considerably when each organization molds and tests its own specimens.

It should be noted that all specimens molded or tested by laboratory D in phase II, series 3, produced abnormally high test values. Apparently some major variation from the prescribed procedure has been introduced. Consequently, the statistical interpretation of results shown in Tables VIII and IX may be misleading due to these values.

Examination of all the test data from both phases indicates that a degree of repeatability of not more than 15 per cent at the maximum may be expected, with an over-all average of some 3 per cent. The degree of reproducibility may be expected to vary by not more than 23 per cent with an over-all average of some 9 per cent. Elimination of the data from laboratory D would reduce the last two figures to 17 and 8 respectively.

## EFFORTS TO IMPROVE COORDINATION AND REPRODUCIBILITY[4]

Shortly after the cooperative testing program was started, representatives of the North Carolina Highway Department visited several of the laboratories involved and observed the technique and procedures used by their technicians. As might be expected, these visits disclosed a diversity of operations that could account for differences in final results. While most of these variables were relatively minor, a combination of several could contribute heavily to the inconsistent results. A number of suggestions were made to standardize the operating procedures for handling and preparation of materials. The variations among laboratories did not appear to improve as a result of these visits.

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[4] Reproducibility refers to values among different laboratories.

At the close of phase I, which included eight separate series, a conference or round-table discussion was held in Raleigh, N. C. to analyze the test data and attempt to establish criteria for better uniformity by elimination of the most obvious differences in procedures. It was also decided to prepare a questionnaire covering each of the details involved in fabrication and testing bituminous mixtures by Method D 1074. Answers to these questions brought out the necessity for describing the method more precisely than it is currently written especially with regard to the blending, mixing and molding of the test specimens. While the method is quite specific on most points, it leaves several of the necessary steps to the option of the technician.

## COMPRESSION TEST USED FOR FIELD STABILITY INVESTIGATIONS

While the compression test may be used quite successfully as a laboratory design tool, no provisions have been made in the development of this method for testing mixtures in the field. All design data are based on mixtures prepared in the laboratory using the raw materials before going through the drier. It is realized that duplication of results by field methods is not an absolute necessity, but it would be desirable to

TABLE X.—LABORATORY DESIGN STABILITIES *versus* FIELD STABILITIES.

This table shows the relationship of specific gravity and stability values of mixtures made in the laboratory using raw materials as compared with an identical mixture molded from the materials mixed in the pug mill at the asphalt plant. Averages of each group are not shown due to the wide variety of aggregate involved, that is, the extreme differences in specific gravity and other physical characteristics.

| Laboratory Mixes | | Field Mixes | | Per Cent Change in Stability |
|---|---|---|---|---|
| Specific Gravity | Stability, psi | Specific Gravity | Stability, psi | |
| H TYPE BINDER (1-IN. AGGREGATE) | | | | |
| 2.289.... | 256 | 2.305 | 348 | +36 |
| 2.243.... | 253 | 2.288 | 215 | −15 |
| 2.347.... | 250 | 2.371 | 257 | +3 |
| 2.347.... | 250 | 2.374 | 315 | +26 |
| 2.358.... | 250 | 2.350 | 308 | +23 |
| 2.315.... | 267 | 2.313 | 363 | +36 |
| 2.408.... | 318 | 2.403 | 395 | +24 |
| 2.261.... | 201 | 2.308 | 368 | +83 |
| 2.279.... | 260 | 2.329 | 278 | +7 |
| 2.280.... | 205 | 2.283 | 278 | +36 |
| 2.322.... | 178 | 2.221 | 228 | +28 |
| 2.323.... | 247 | 2.316 | 279 | +13 |
| 2.237.... | 266 | 2.286 | 304 | +14 |
| 2.324.... | 268 | 2.323 | 220 | −18 |
| F-2 WEARING SURFACE (3/4-IN. AGGREGATE) | | | | |
| 2.304.... | 305 | 2.281 | 288 | −6 |
| 2.335.... | 348 | 2.221 | 228 | −34 |
| 2.338.... | 314 | 2.296 | 313 | 0 |
| I TYPE WEARING SURFACE (3/4-IN. AGGREGATE) | | | | |
| 2.293.... | 346 | 2.310 | 340 | −2 |
| 2.303.... | 340 | 2.288 | 407 | +20 |
| 2.315.... | 321 | 2.321 | 283 | −12 |
| 2.251.... | 265 | 2.286 | 300 | +13 |
| 2.348.... | 277 | 2.332 | 251 | −9 |
| 2.369.... | 302 | 2.329 | 315 | +4 |
| 2.338.... | 364 | 2.316 | 397 | +6 |
| 2.312.... | 390 | 2.361 | 426 | +9 |
| 2.342.... | 343 | 2.355 | 358 | +4 |
| 2.309.... | 323 | 2.363 | 444 | +37 |
| 2.255.... | 253 | 2.330 | 408 | +61 |
| 2.329.... | 331 | 2.303 | 314 | −5 |
| 2.292.... | 232 | 2.282 | 298 | +28 |
| 2.323.... | 309 | 2.330 | 351 | +14 |
| 2.312.... | 254 | 2.286 | 298 | +17 |
| 2.270.... | 310 | 2.237 | 330 | +6 |
| 2.307.... | 320 | 2.361 | 325 | +2 |
| 2.298.... | 329 | 2.301 | 290 | −12 |
| 2.255.... | 326 | 2.288 | 291 | −11 |
| 2.312.... | 403 | 2.258 | 504 | +25 |
| 2.280.... | 300 | 2.262 | 235 | −22 |
| 2.313.... | 254 | 2.291 | 310 | +22 |
| 2.310.... | 283 | 2.321 | 325 | +15 |
| 2.278.... | 307 | 2.278 | 296 | −4 |

have an approved method for making such tests of the stability and other characteristics.

All tests performed in the following field study and the results derived therefrom were for research purposes only and were not used as a means of control of quality of paving projects.

The investigation was made during the 1955 construction season and included all types of bituminous concrete mixtures used in North Carolina. Two different methods were employed in preparation of the test specimens:

1. At a number of asphalt plants within three hours distance of the central laboratory, a sample of the hot mixture was taken, placed in an insulated box and delivered quickly to the laboratory where specimens were molded. Each sample contained approximately 200 lb of mixture. It was found that the temperature of the mixture did not drop more than 25 to 30 F during a 3-hr period.

2. At those plants located beyond a 100-mile radius of Raleigh, molds and forming equipment were carried to the plant where specimens were made using plant-mixed paving mixtures. The specimens were placed in a specially designed ice box and, while chilled, returned to the laboratory where they were cured and broken in the prescribed manner.

A careful examination of the results shows little or no difference between the two "field" methods. As long as the specimens are molded at the proper temperature without reheating the mixture it apparently makes no difference whether they are made in the laboratory, as described in method 1 above, or in the field, as described in method 2 above. When the mixture was brought to the laboratory for specimen preparation, the entire amount for the test came from the same batch. When the samples were made in the field, approximately five different batches were represented in each test.

The total amount of test data accumulated in the investigation is too voluminous to be included in this report. The data have been condensed into Table X. There are two factors that should be kept in mind when reviewing these results. First, the field values of specific gravity and stability represent the average of from 10 to 50 samples for each project. Many of the projects were of such short duration that only one field check could be made, hence only 10 specimens were made from that particular project. On the larger jobs, several visits resulted in more samples being taken. A second factor to be kept in mind is the day-to-day variation of the gradation of the aggregate in the mixtures. This factor would have some bearing on final results. In general, the composition of the mixtures did not exceed the tolerances permitted by the specifications, so excessive variations could not be attributed to non-specification proportions.

The data shown in Table X compare values obtained in the laboratory on design mixes with the average of all checks made in the field for each type of paving mixture. Previous tests in the North Carolina laboratory have indicated a degree of repeatability of 5 per cent or less on design mixes when using the same materials. Slight variations may be due to differences in the individual operator's technique. These individualities among different personnel would not account for some of the large variations which appeared on some projects, however.

The H type binder showed an average increase in stability of 21 per cent over the laboratory design mixes, the I type surface mixtures showed an average increase of 9 per cent, while the F-2 wearing surface showed an average decrease of 13 per cent below the stability of the laboratory design mixes. The comparisons apparently follow no uniform pattern of increase or decrease in stability after the aggregates have gone through the plant drier.

Only one type of mixture, the I type, was within the 10 per cent average range considered acceptable for control work of this nature. Even then, examination of the individual averages for this type of mixture shows that 14 out of 24 projects were outside of the 10 per cent range.

These results emphasize the need for uniformity of procedure in having stability determinations made either on the raw aggregates before going through the drier or on the dried, processed materials actually prepared for use in the specific project. Apparently some change takes place in the physical characteristics of the aggregates during the drying, screening, and batching operations which may, and usually does, cause a change in the final stability value.

## CONCLUSIONS

The conclusions formed as a result of the described investigation may well apply to other tests of a similar nature. We believe the following items to be the most significant conclusions derived from the cooperative study:

1. The repeatability[5] of Method D 1074 may be as high as 15 per cent but should average 3 to 5 per cent.

2. The reproducibility[4] of Method D 1074 may be as high as 23 per cent but should average 9 or 10 per cent.

3. Any stability requirements based on Method D 1074 should be determined with the raw aggregates, that is, aggregates that have not been processed at the asphalt plant. Some change takes place in the physical characteristics of the aggregates when they go through the plant drier. This change will sometimes result in an increase in stability while in other cases it will result in a lower stability.

4. Method D 1074 should be more explicit in detail. It is apparent that the sum total of the minor detailed operations left to the discretion of the individual technician has a definite bearing on final results. Therefore it is essential that as many operational procedures as possible be described in minute detail, leaving a minimum of option for the technician.

The main reasons for the discrepancies obtained are as follows:

(*a*) Different methods employed for blending and heating aggregates and asphalt for specimen preparation.

(*b*) Variations in the application of heat during the mixing operation.

(*c*) Variations in type 77 F air bath and methods employed to bring specimens to proper test temperature.

(*d*) Lack of uniformity in adjusting rate of travel of compression head for application of load.

Due to the several variables involved it was impossible to isolate any one item as being the chief contributor to the variations.

It is believed that the handling and preparation of aggregates for molding test specimens should be standardized. The drying, sieving, reproportioning, heating, addition of bitumen, and mixing should be the same in each case, up to the point of actually molding the test specimens. At present each laboratory follows a somewhat different procedure in preparing the aggregate materials.

As a result of the cooperative tests, certain revisions for Method D 1074 – 55 T have been suggested and this matter has been taken under advisement by Subcommittee B-2 of Committee D-4.

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[5] Repeatability refers to values within the same laboratory.

*Acknowledgment:*

The authors would like to express their appreciation to all laboratories who took part in the described coöperative study. These laboratories are as follows:

Virginia Department of Highways Laboratory, Richmond, Va.
Parker Laboratory, Charleston, S. C.
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Froehling and Robertson Laboratory, Richmond, Va.
Bureau of Public Roads, Physical Research Branch, Washington, D. C.
National Crushed Stone Assn. Laboratory, Washington, D. C.

Special appreciation is also extended to Mr. R. S. Jivatode, Research Analyst, who made the statistical interpretation of the accumulated data.

# THE ROLE OF PIGMENTS IN THE WEATHERING OF AUTOMOTIVE PAINTS*

By S. B. Twiss,[1] W. L. Weeks,[1] and F. O. Thomas[1]

The reactions occurring during the weathering of paints are exceedingly complex, but it is well recognized that degradation of the vehicle is the primary cause of paint film breakdown. The interaction of the vehicle and pigments in the film are undoubtedly important, since different pigments produce different weathering defects with the same vehicle system. Whether these different weathering effects are related to the pigment-vehicle interaction is not known, and relatively little work has been reported in the literature along these lines. The purpose of the present work was to attempt to correlate certain properties of pigments with the defects occurring during weathering of paints containing these pigments. As a start on the problem, the light and electron microscopes were used as the primary tools of investigation.

Due to the importance of vehicle breakdown in paint weathering, a number of workers have investigated this aspect of paint failure, which is basic to any understanding of the effects of pigments in weathering. Fitzgerald (**1**),[2] studying alkyd enamels, found that degradation begins with oxidation of the unsaturated bonds in the drying oil during the air drying or baking stage and spreads to the glyceryl-phthalate part of the alkyd. Ultimately, volatile products are produced resulting in a reduction in film thickness and shrinkage. Crecelius *et al* (**2**), using infrared spectrophotometry, confirm the work of Fitzgerald. They find that loss of weight by the film during ultraviolet exposure is highest during the first six hours. After 34 hr, the films were practically consumed.

Fitzgerald (**1**) found that when a film is exposed to sunlight, the longer wavelengths penetrate deeper than the shorter ones. These bulk film photolytic reactions are more gradual than those initiated by short wavelengths near the surface. As a result of bulk reactions, shrinkage, increase in density, embrittlement, and yellowing occur in the film. Miller (**3**), using mercury lamps as radiation sources, found that short wavelengths in the ultraviolet caused surface degradation while longer wavelengths (greater than 350 m$\mu$) produced reactions in the bulk film. Similarly, Gusman and Stroupe (**4**), using both pigmented and unpigmented films, found short wavelength ultraviolet light to be most active for photochemical deterioration. They note also that, due to the filtering action of the atmosphere, wavelengths below 300 m$\mu$ are unavailable for photochemical action. Marshall (**5**) noted that short wavelengths are most effective in reducing tensile

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* Presented at a meeting of Committee D-1 held at Atlantic City, June 18, 1957.

[1] Chrysler Corporation, Engineering Division, Detroit, Mich.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1365.

strength of free unpigmented paint films, with a combination of long wavelength ultraviolet and near infrared being somewhat less effective. The latter combination of wavelengths was shown to be most effective in reducing elongation.

The presence of pigments has been found by some studies to affect vehicle breakdown. A number of investigators, using ultraviolet light, have studied the action of pigments in paint films. Fitzgerald (**1**) has determined the rate of gas evolution in the bulk film as opposed to the surface during exposure to very short ultraviolet light. The rate of gas evolution from a film incorporating 20 per cent by weight of titanium dioxide was shown to be 20 per cent slower than for a clear film indicating that this pigment is merely an inert space filler. According to Elm (**6**), the progressive loss in film thickness during exposure can be reduced by some pigments (notably zinc oxide is effective in reducing chalking by virtue of its high ultraviolet absorption coefficient). Wartman (**7**), using titanium dioxide pigment in vinyl chloride resins, demonstrated that the evolution of hydrogen chloride as a result of weathering was restricted to the upper layers, although it occurred practically unreduced in the bottom layers of an unpigmented film. Gusman and Spell (**8**) concluded that some pigments function to restrict photolytic reactions to the upper layers of the paint film. Kronstein (**9**) mentions evidence of severe cracking in an alkyd resin pigmented with calcium carbonate (which possesses poor hiding power) when the film was exposed for 400 hr in a twin arc type D weatherometer[3] without water spray. He suggested that a pigment which transmits well in the ultraviolet affords little protection for the vehicle.

Work with the light microscope on paint weathering has not been extensive. Subsurface light scattering by the paint film makes it difficult to resolve the smaller pigments and the fine structure resulting from degradation. Most light micrographs in the literature are at magnifications below 200 ×.

Investigations of weathering of paint films using the electron microscope have not been numerous. As long ago as 1947, Tilleard and Birbeck (**10,11**) developed replication techniques for paint surfaces and noted the increase in roughness of paint films upon exposure to accelerated weathering cycles. Kienle and Maresh (**12**), using the electron microscope to study pigments, thin sections, and pigmented surfaces, indicated that a large number of factors can affect the appearance of a pigmented film including steps in the formulation and drying. Their micrographs of chalked and bronzed surfaces show a difference in appearance of surface details between the two types of degradation. Smith (**13**), also studying pigments, surfaces, and thin sections, showed exudates, types of blooming, and progressive surface roughening accompanying pigment release as a paint degrades both from natural weathering and exposure to ultraviolet radiation alone.

In a previous paper by Twiss, Teague, and Weeks (**14**), work in our laboratories was reported on the development of suitable replication techniques for the electron microscope study of paints. The application of these methods to the study of the degradation of several chalking type paints by natural weathering and accelerated ultraviolet exposure was presented. This early work indicated that a freshly painted surface is microscopically smooth with pigment particles near the surface covered with a thin film of vehicle. Exposure to ultraviolet radiation progressively degrades the organic vehicle resulting in a loss of vehicle and shrinkage. This effect exposes pigment particles lying near the surface, and these loose particles are a major factor contributing to the loss of gloss associated with chalking. Natural weathering was shown to produce a far rougher surface than accelerated ultraviolet exposure.

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[3] Tentative Recommended Practice for Operation of Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Artificial Weathering Test (E 42 – 55 T), 1955 Book of ASTM Standards, Part 4, p. 1311.

The XW Weatherometer manufactured by Atlas Electric Devices, Inc., Chicago, Ill., corresponds to Type E weatherometer in this standard.

TABLE I.—CHALKING TYPE PIGMENTS.

| Pigment | Composition | Concentration, per cent | Gloss | Chalk | Bronze | Check |
|---|---|---|---|---|---|---|
| No. 1. Titanium Dioxide.... | Inorganic<br>Rutile $TiO_2$ | 35 | 24 | Medium + | ... | ... |
| No. 2. Red Iron Oxide...... | Inorganic—Red Oxide<br>(99 per cent $Fe_2O_3$) | 20 | 13 | Medium + | ... | ... |
| No. 3. Indian Red.......... | Inorganic<br>Iron Oxide (99 per cent $Fe_2O_3$) | 25 | 2 | Bad | ... | Trace |
| No. 4. Ferrite Yellow....... | Inorganic<br>Yellow Iron Oxide (86 per cent $Fe_2O_3$) | 30 | 19 | Medium + | ... | ... |
| No. 5. Lamp Black.......... | Furnace Black (1 per cent volatile) | 5 | 16 | Medium + | Slight spot | ... |
| No. 6. Copper Maroon...... | Metallo-organic<br>Cuprous Ferricyanide | 20 | 15 | Medium + | ... | ... |

Complementing a study by Gerson (**15**) on the mechanism of paint film breakdown, Twiss, Teague, and Weeks (**16**) investigated by electron microscopy the degradation of light green and dark maroon enamel and lacquer subjected both to natural weathering in Florida and to accelerated testing in an XW (type E)[3] type weatherometer. It was found that the light green enamel and lacquer followed a pattern of weathering similar to that hypothesized in the first paper; vehicle degrades and shrinks at the surface releasing pigment particles and giving rise to a typical chalked appearance. The loss of gloss accompanying the weathering of dark maroon paints (98 per cent toluidine maroon, 2 per cent carbon black pigment), however, was found to be due principally to the development of deep fissures throughout the paint which is characteristic of checking. In accord with Gerson's work, electron microscopy showed little correlation in appearance between natural and XW weatherometer exposure for the maroon paints. Loss of gloss due to natural weathering was found to be far more severe than was the case for artificial exposure. In the case of XW weatherometer results, the electron microscope showed that thin sheets of degraded vehicle developed on the surface of the maroon lacquer. These sheets interfered little with gloss and could be removed to reveal a slightly roughened surface underneath. The results suggest that the pigment in the dark maroon paint did not afford as much protection for the underlying vehicle from the standpoint of photochemically active energy as did the pigment contained in the light green

TABLE II.—BRONZING TYPE PIGMENTS.

| Pigment | Composition | Concentration, per cent | Gloss | Chalk | Bronze | Check |
|---|---|---|---|---|---|---|
| No. 1. Iron Blue<br>Chinese Blue | Inorganic<br>Iron Ferroferricyanide | 20 | 44 | Trace | Medium + spot | |
| Milori Blue | Inorganic<br>Iron Ferroferricyanide | 20 | 64 | Trace | Medium spot | |
| No. 2. Gold Paste | Inorganic<br>Ferric Hydrate | 10 | 59 | Trace + | Medium | |
| No. 3. Thio Fast Red | Organic<br>Thio Indigo | 25 | 58 | Slight | Slight | Slight + |
| No. 4. Carbon Black | Carbon Black | 5 | 93 | Slight | Slight + spot | |
| No. 5. Burnt Sienna | Inorganic<br>Iron Oxide—Natural Earth (60 per cent $Fe_2O_3$) | 20 | 40 | Slight + | Medium spot | |
| No. 6. Chrome Green | Inorganic<br>Lead Chromate 75 per cent<br>Iron Blue 25 per cent | 20 | 43 | Slight | Medium spot | |
| No. 7. Amberine Maroon | Organic<br>Toluidine Maroon<br>(Naphthol or Arylamide) | 25 | 66 | Slight | Slight + spot | Slight + spot |
| No. 8. Maroon Gold | Metallo-organic<br>Cuprous Ferricyanide | 25 | 53 | Slight + | Slight spot | |
| No. 9. Phthalocyanine Blue | Metallo-organic<br>Copper Phthalocyanine | 10 | 67 | Trace | Medium + spot | |

TABLE III.—CHECKING TYPE PIGMENTS.

| Pigment | Composition | Concentration, per cent | Gloss | Chalk | Bronze | Check |
|---|---|---|---|---|---|---|
| No. 1. Clear Vehicle<br>Clear Florida | Organic | ... | 4 | ... | | Very bad |
| Clear Detroit | Organic | ... | 64 | ... | | Medium |
| No. 2. Caddy Red | Metallo-organic<br>Pyrazolone Red<br>(Calcium $\beta$-oxynaphthoate) | 20 | 16 | Medium | ✓ | Bad micro |
| No. 3. Organic Orange | Organic<br>Dinitraniline Orange | 25 | 9 | Medium + | | Trace polish |

TABLE IV.—COMBINATION TYPE PIGMENTS.

| Pigment | Composition | Concentration, per cent | Gloss | Chalk | Bronze | Check | Electron Micrograph rating |
|---|---|---|---|---|---|---|---|
| No. 1 Molybdate Orange | Inorganic<br>Lead Chromate 60 per cent<br>Lead Sulfate 20 per cent<br>Lead Molybdate 10 per cent + | 35 | 50 | Slight | Medium spot | . . . | Chalking and Bronzing |
| No. 2. Chrome Yellow | Inorganic<br>Lead Chromate 70 per cent<br>Lead Sulfate 30 per cent | 40 | 49 | Slight | Medium<br>Water spot | . . . | Chalking and Bronzing |
| No. 3. Toluidine Red | Organic<br>$\beta$-naphthol diazo<br>$m$-nitro-$p$-toluidine | 25 | 66 | Slight | Slight + | Slight + | Chalking and Bronzing |
| No. 4. Chrome Orange | Inorganic<br>Lead Chromate 57 per cent<br>Lead Sulfate 43 per cent | 40 | 27 | Medium + | Slight<br>Water spot | Bad Micro | Chalking, Bronzing and Checking |
| No. 5. Phthalo Green | Metallo-organic<br>Chlorinated Copper<br>Phthalocyanine | 10 | 68 | Slight | Medium | Bad Figure and Polish | Bronzing and Checking |
| No. 6. Green Gold | Metallo-organic<br>Nickel Complex of<br>Azo Yellow | 10 | 49 | Trace | Slight | Slight Figure<br>Bad Polish | Bronzing and Checking |
| No. 7. Indo Blue | Indanthrene Blue Lake<br>(37 per cent $Al_2O_3$, ashed) | 10 | 48 | Medium | Slight | Slight<br>Polish | Bronzing and Checking |
| No. 8. Indo Orange | Indanthrene Orange Lake | 5 | 12 | Slight | | Bad Figure and Polish | Bronzing and Checking |
| No. 9. Chrome Hydrate Green | Inorganic<br>Chromium Hydroxide<br>(83 per cent $Cr_2O_3$) | 10 | 7 | Slight | Medium spot | Bad Micro | Bronzing and Checking |
| No. 10. BON Red | Metallo-organic<br>Manganese $\beta$-oxynaphthoate | 20 | 51 | Slight | Slight + | Slight + | Bronzing and Checking |

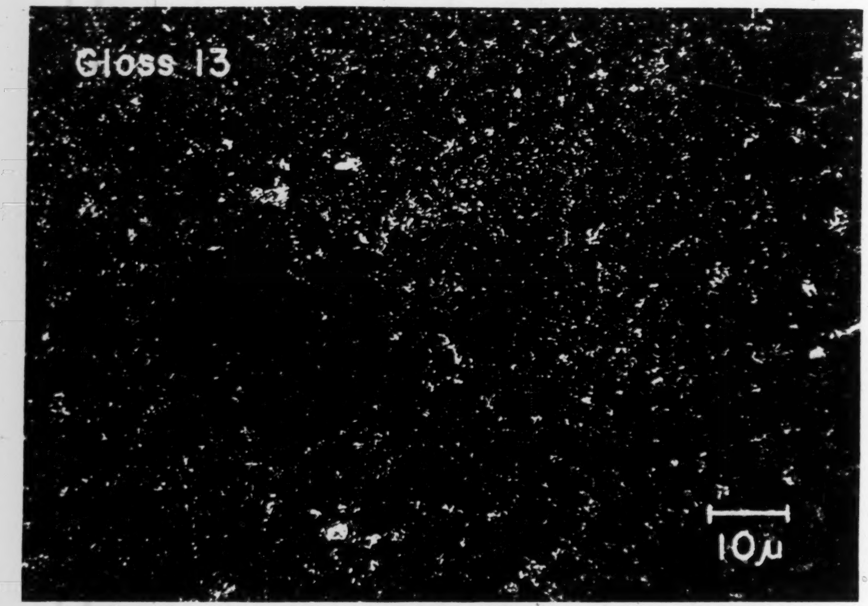

FIG. 1.—Red Oxide, Light Micrograph, Aluminized Surface, Gloss 13.

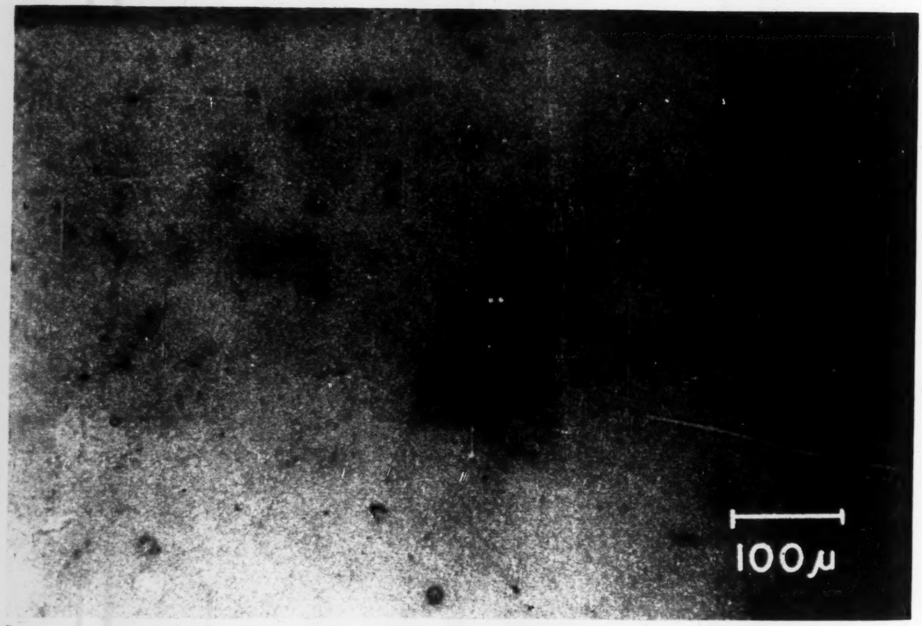

FIG. 2.—Titanium Dioxide, Light Micrograph, not Aluminized.

paint (95 per cent titanium dioxide, 5 per cent phthalocyanine green).

## EXPERIMENTAL

For the purpose of this investigation, 28 different pigments were studied. These pigments represent most of those commonly used in automotive finishes. The vehicle for all the paints was a standard 90 per cent alkyd - 10 per cent melamine resin. The test panels were prepared according to standard automotive paint practices, the finishes being sprayed on bonderized steel plates. The panels had been exposed for one year in southern Florida, and subsequently the type and severity of the degradation characterizing each panel were noted visually. Gloss ratings were also determined for each of the weathered paints using a Gardner 60-deg glossmeter. These visual and gloss ratings are listed in the descriptive tables (Tables I, II, III, and IV) following the discussion of each type of degradation studied.

The uncompounded pigments were obtained in either a dry form or as a vehicle-pigment paste. These pigments were examined with the electron microscope to establish the pigment particle size, shape, and other characteristic data. The uncompounded pigments and the weathered panels, together with visual ratings and gloss readings on the weathered panels, were supplied by the Ditzler Color Division of Pittsburgh Plate Glass Co.

In the early stages of the light microscope work, it was found in several instances that interpretation of the surface structure was greatly enhanced if the panels being examined were first coated with a thin film of aluminum by vacuum evaporation. Without the metallic coating, the surface and immediate subsurface of the panel diffuses light yielding a flat micrograph. With a thin film of highly reflecting metal, the surface contours are not distorted, but the contrast in the resulting micrograph is increased substantially. Figures 1 and 2 are micrographs of chalked surfaces; the surface shown in Fig. 1 has been aluminized while that shown in Fig. 2 has not. It can be seen that the process permits easier interpretation of the micrograph. When the weathered paint exhibited "bronze spot" (Fig. 18), aluminizing was found to destroy the iridescence associated with the defect. In this case, contrast was obtained by the use of oblique illumination. The light micrographs were obtained using a Bausch & Lomb research type microscope in conjunction with an L camera.[4] The source of illumination was a carbon arc. A green, solution type filter was placed between the sample and the light source.

Poly(vinyl alcohol) - silica replicas shadowed with chromium were used for the electron microscope study of the weathered paint surfaces. The replication technique is identical to that used in previous studies (**14,16**). The dry pigment samples were prepared for study by grinding in a suitable solvent with a mortar and pestle. Using this technique, some pigments dispersed well in water using Aerosol OT as a wetting agent. Other pigments gave better dispersions in xylene using Raybo 6 as a dispersant. The thick paste obtained after grinding was diluted with solvent until the dispersion was sufficiently thin to permit examination at 15,000 × magnification. For those pigments originally obtained in a paste form (green gold, toluidine red, and phthalocyanine green and blue) no grinding was employed. They were merely diluted with xylene and subsequently handled like the dry pigments. The uncompounded pigment specimen

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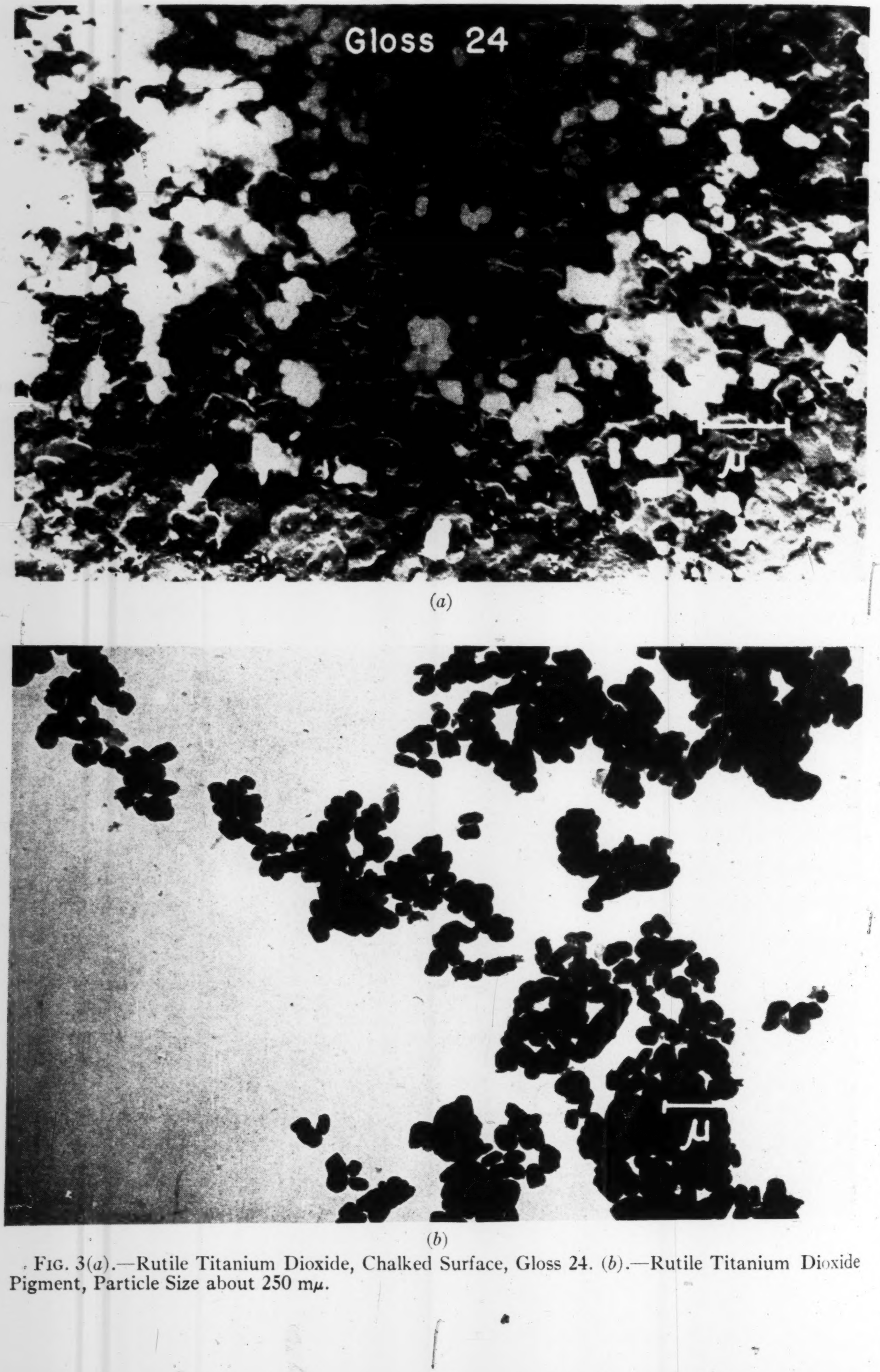
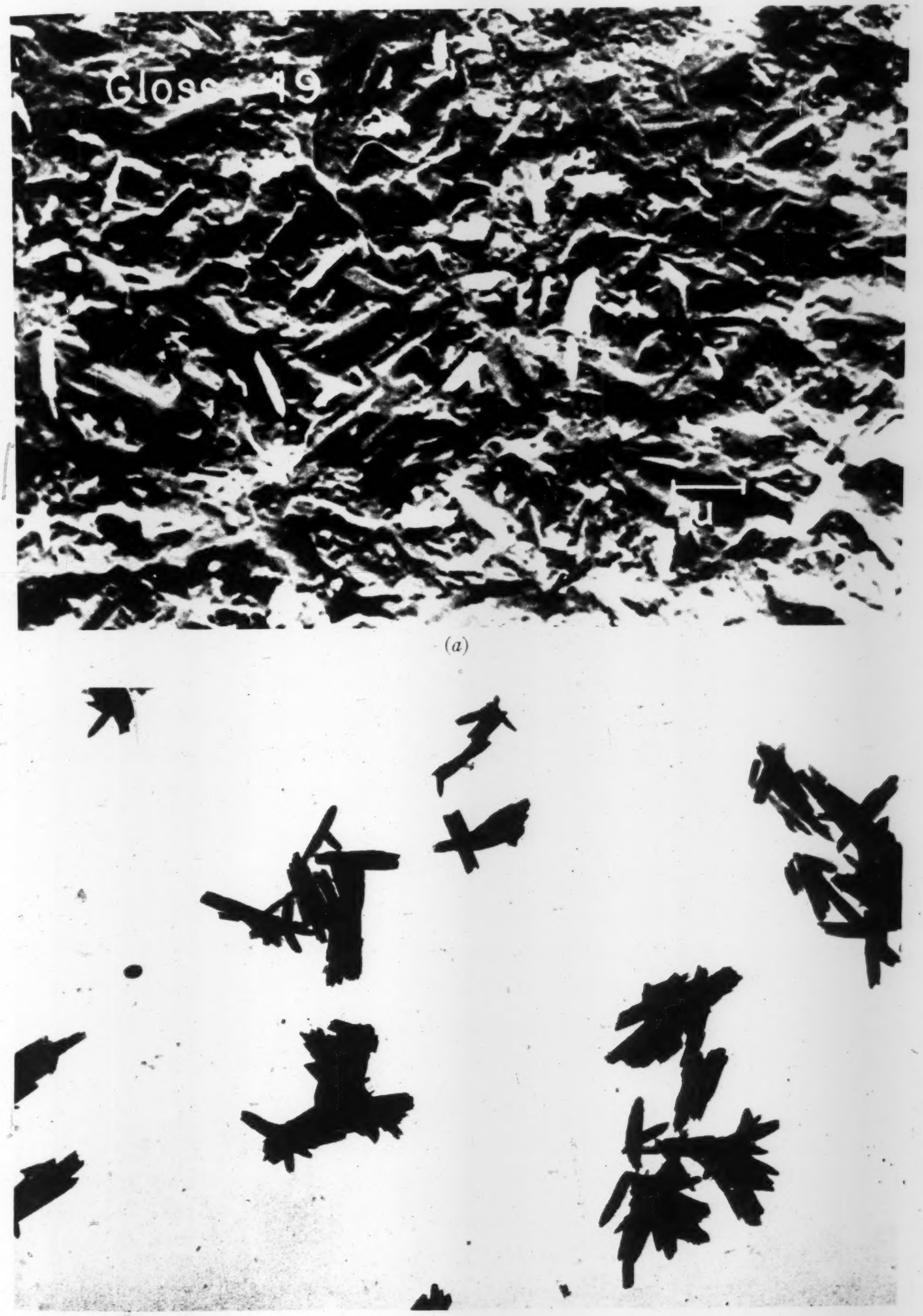
[4] A Bausch & Lomb Model CM microscope (B. & L. Cat. No. 31-21-66-31) in conjunction with a Bausch & Lomb Model L camera (B. & L. Cat. No. 42-14-42).



(*a*)

(*b*)

FIG. 3(*a*).—Rutile Titanium Dioxide, Chalked Surface, Gloss 24. (*b*).—Rutile Titanium Dioxide Pigment, Particle Size about 250 $m\mu$.



(a)

(b)

FIG. 4(*a*).—Ferrite Yellow, Chalked Surface, Gloss 19. (*b*).—Ferrite Yellow Pigment, Particle Size about 50 by 600 m$\mu$.

was prepared by drying a drop of the dilute dispersion on a thin parlodion substrate.

The microscope used was a Radio Corp. of America model EMU-2, and the shadowing was conducted between $10^{-4}$ and $10^{-5}$ mm of mercury pressure in a vacuum evaporator constructed in our laboratory. To obtain a "positive" picture of the paint surface obtained with a two-stage replication procedure, the negatives taken with the electron microscope were reprinted as positives and final prints made from the latter. This procedure gives correct rendition of height, depth, and shadows but changes the pigment which transferred from the paint surface to the replica from opaque black to white particles. Micrographs taken of pigment dispersions themselves show the particles properly as opaque to the electron beam and hence black.

## CHALKING TYPE PIGMENTS

Visually, chalking is characterized by a severe dulling of the paint surface. When the surface is wiped with a finger, pigment can be easily removed, indicating that a certain amount of pigment has been released at the film-air interface during degradation.

A preliminary survey of several chalking type surfaces was taken with the light microscope. After the surfaces had been metallized with aluminum to increase reflectivity, severe light scattering by the rough surface was revealed. The light micrograph of Fig. 1 shows tiny pinpoints of light alternating with very dark adjacent areas. The points of light represent pigment particles and those vehicle areas oriented with their surfaces normal to the incident beam of light. It has been observed that the greater the severity of chalking the greater is the contrast seen in these light micrographs. However, the resolving power and magnification obtainable with the light microscope is insufficient to reveal details of the surface appearance, nor can the extent of pigment release or the size and shape of the pigment be accurately determined.

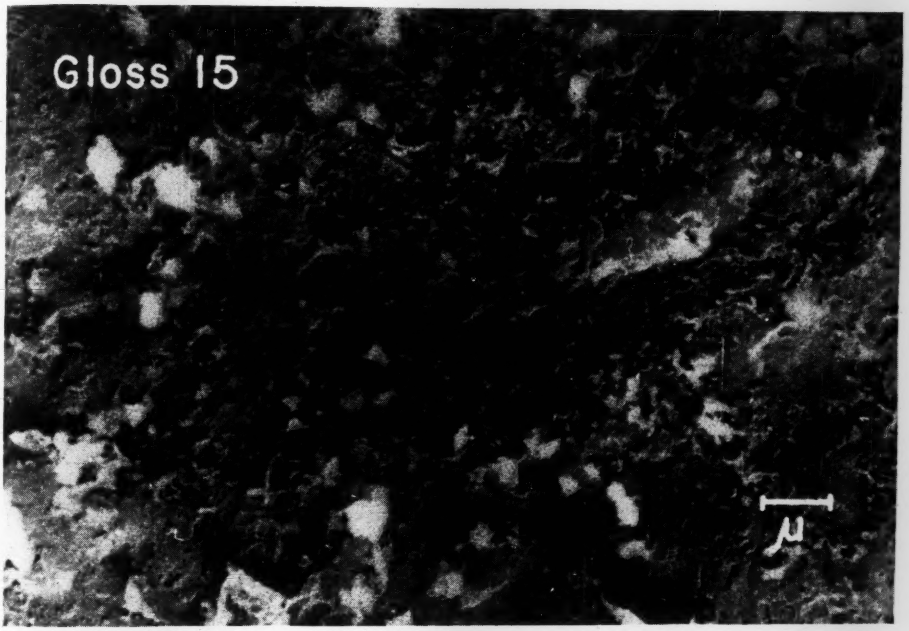
The electron microscopic appearance of the surface of chalked paints has been fairly well established. Titanium dioxide is typical of a pigment which promotes chalking. A micrograph of an exposed paint containing 35 per cent by weight of titanium dioxide is shown in Fig. 3(*a*). The micrograph reveals considerable shrinkage of the film surface accompanied by the complete release of a number of pigment particles.

This pigment, released by weathering and lifted from the surface during replication, appears as single white particles or clumps of particles in the micrograph. Many partially exposed pigment particles are visible in the surface which has assumed a severely roughened appearance during exposure. A small amount of semiopaque material, presumed to be partially decomposed vehicle, can be seen scattered among the freed particles.

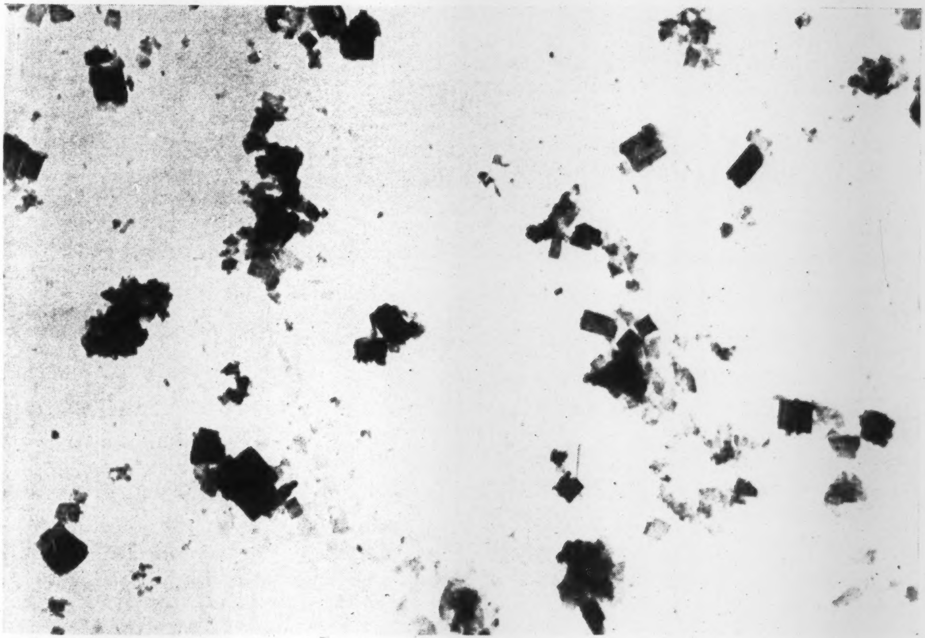
When the uncompounded pigment particles were examined with the electron microscope (Fig. 3(*b*)), they appear as opaque particles identical in size and shape to the free pigment on the degraded paint surface. At this magnification, 10,000 ×, they are slightly oval or rectangularly shaped particles about 250 $m\mu$ across.

A chalked surface is again encountered in a paint incorporating another inorganic pigment, ferrite yellow (Fig. 4(*a*)). The pigment is present as distinctly rod-shaped or acicular particles. When released from or exposed on the surface by vehicle erosion and degradation, the pigment leaves distinctive, large slots at the surface which are capable of reducing gloss severely. Loose particles are not as abundant here as is the case with titanium dioxide.

Comparing the dry pigment in Fig.



(a)



(b)

FIG. 5(a).—Copper Maroon, Chalked Surface, Gloss 15. (b).—Copper Maroon Pigment, Particle Size—about 200 by 400 mμ

4(*b*) with the needle shaped particles released by the weathered film, it is clear that weathering has resulted in no change in either the size or the shape of the pigment. Some of the particles appear to be bundles of smaller rod-shaped crystals which apparently easily agglomerate to function as single larger particles.

Copper maroon (Fig. 5(*a*)) is an example of a metallo-organic pigment which, according to a visual rating, chalks. Electron microscopy reveals a generally roughened surface with a moderate number of discrete pigment particles scattered over the surface. Many more pigment particles lie partially exposed at the surface contributing to large irregularities in surface contour and a reduction of gloss to 15. The micrograph also illustrates that chalking is not confined to inorganic pigments.

The uncompounded copper maroon pigment particles (Fig. 5(*b*)) are unique in this study. As shown in the micrograph, they have a characteristic rectangular shape. Large numbers of particles are relatively transparent to the electron beam indicating thin, flakelike shapes. In general, the shape and size of the particles visible in the weathered paint film correspond to the dimensions of the original pigment.

Examination of the above formulations suggests a definition of chalking from the viewpoint of microscopy: chalking consists of the progressive shrinkage and erosion of a paint film surface resulting in the exposure and release of discrete pigment particles and small clumps of particles with the creation of a surface whose roughness is related to the effective pigment size and whose morphology is determined by pigment shape and effective size. The term effective particle size is used here to include pigment agglomerates which, from the standpoint of surface roughness, function as single particles.

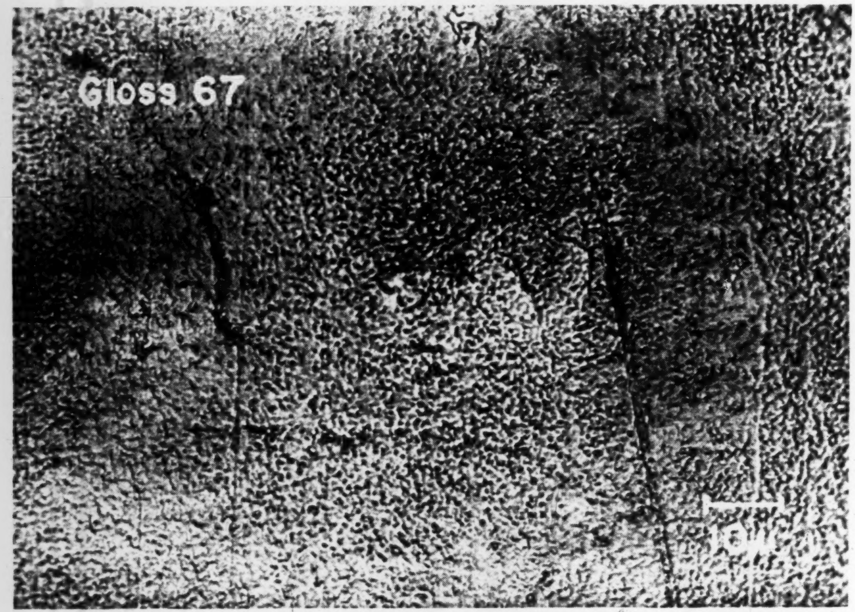
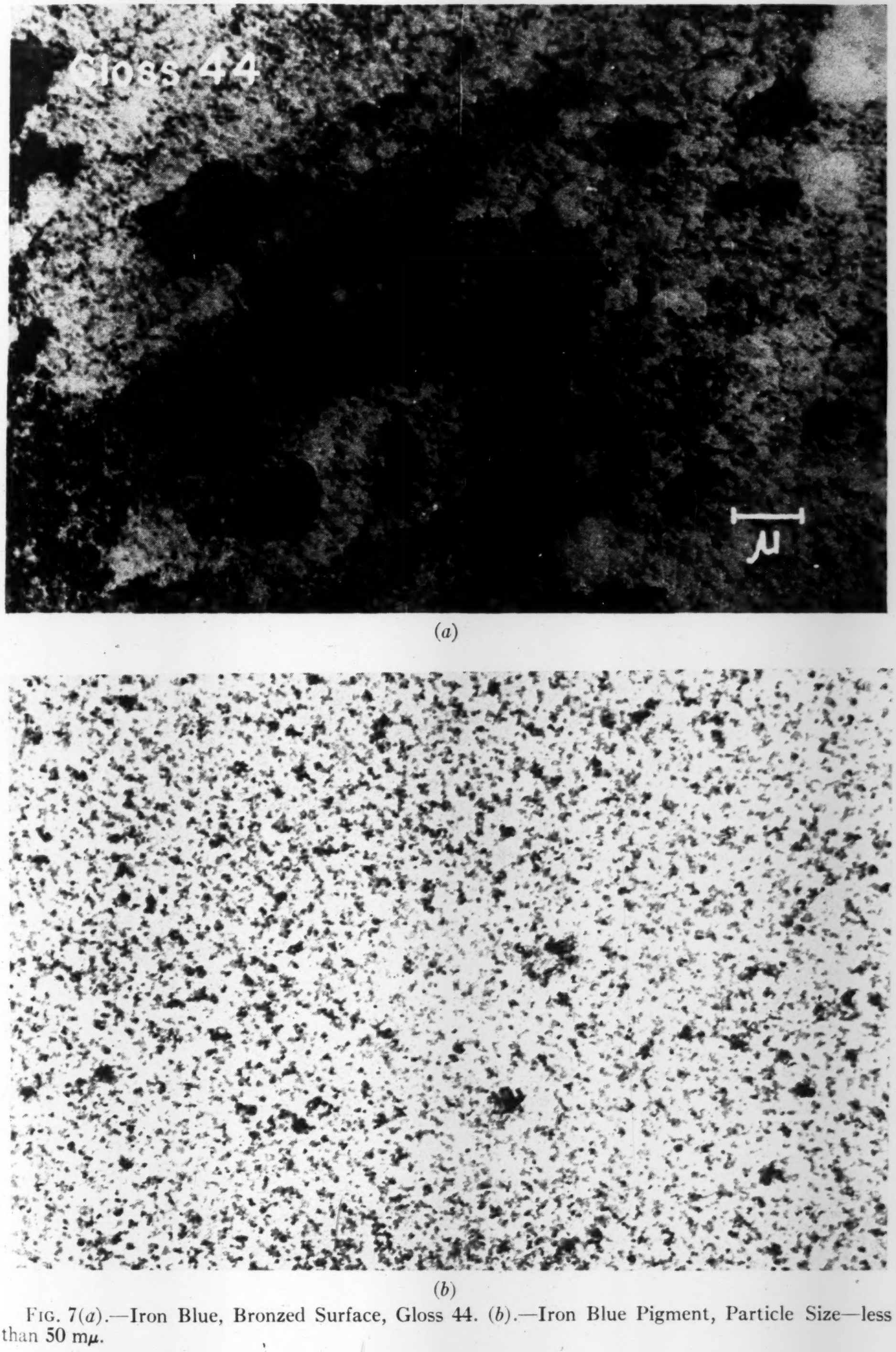


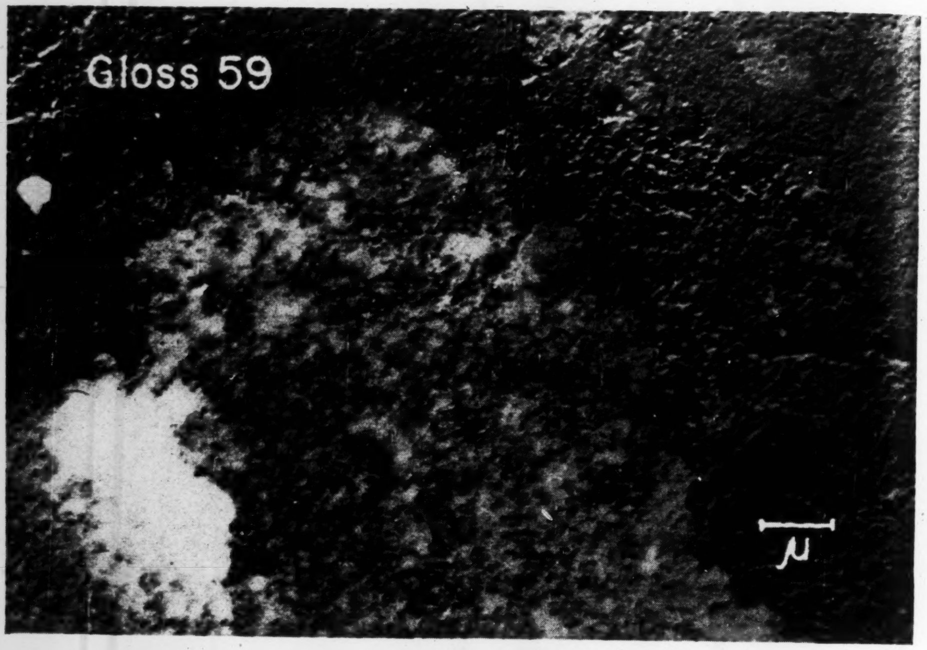
FIG. 6—Phthalocyanine Blue, Light Micrograph, Aluminized Surface, Gloss 67.



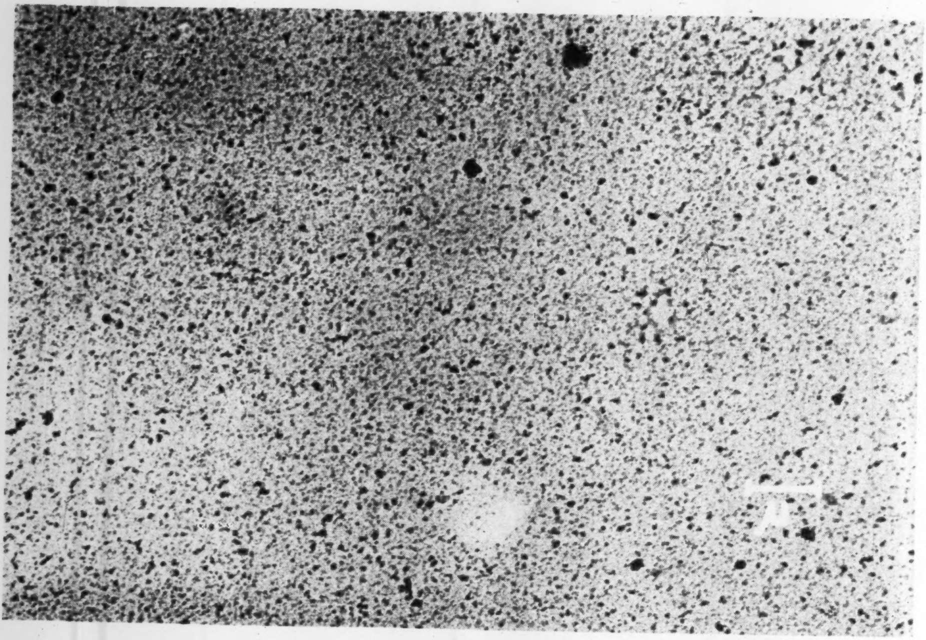
(*a*)

(*b*)

FIG. 7(*a*).—Iron Blue, Bronzed Surface, Gloss 44. (*b*).—Iron Blue Pigment, Particle Size—less than 50 mμ.



(a)



(b)

FIG. 8(a).—Gold Paste, Bronzed Surface, Gloss 59. (b).—Gold Paste Pigment, Particle Size—less than 50 mμ.

Other pigments which showed primarily chalking characteristics were red iron oxide, Indian red, and lampblack. All six chalking pigments listed in Table I are arranged in decreasing order of resemblance to a typical chalked paint as characterized by the above definition. Other pertinent data is included in the table, including the visual ratings of the panels. There is good agreement between the chalk rating as revealed qualitatively by the microscope and as determined by visual examination.

### BRONZING TYPE PIGMENTS

Visual examination of a typical bronzed paint will reveal that a color change has occurred. The paint surface assumes a brownish or purplish tone. Areas of iridescence, similar in appearance to an oil film on a water surface, are generally visible.

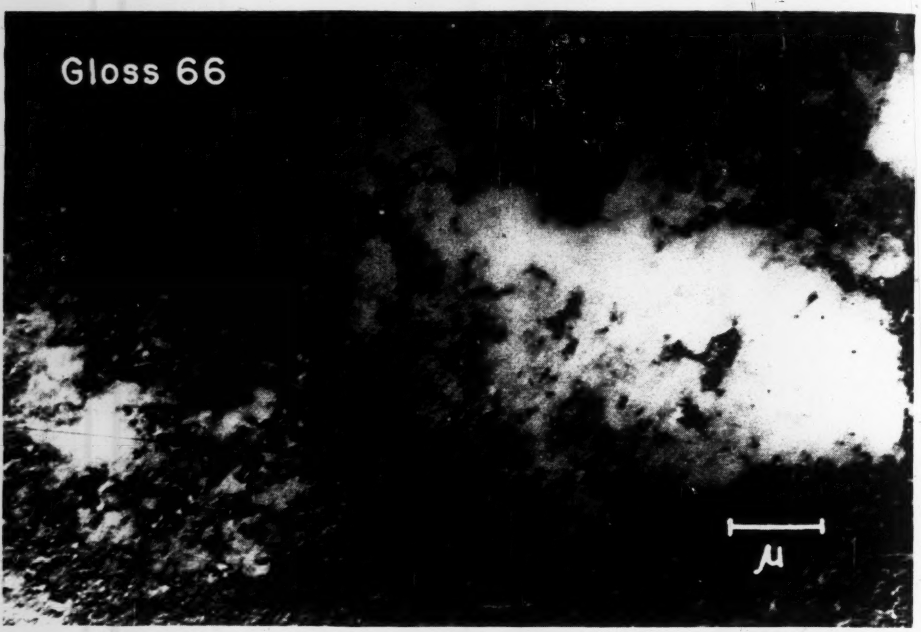
Light micrographs of bronzed surfaces do not present as clearly typical an appearance as those of chalked panels. The smoother surface of aluminized bronzed panels permits very high reflectivity. Figure 6 shows the typical bronzed surface of a phthalocyanine blue pigmented panel. Aside from occasional scratches caused by handling, the micrograph shows a pattern of slight undulations of varying contrast. Compared with typical chalking (Fig. 1), bronzing appears to be characterized by a smoother surface and higher reflectivity. The light microscope is unable to reveal any structure which can be ascribed to pigment and, therefore, it is inadequate for determining the pigment's part in degradation.

Except for the electron micrographs of Kienle and Maresh (**12**), no electron micrographs illustrating this type of degradation were found in the literature. The microscopic appearance of bronzing is very distinctive and quite unlike that of chalking. The typical appearance of bronzing is illustrated in Fig. 7(*a*).

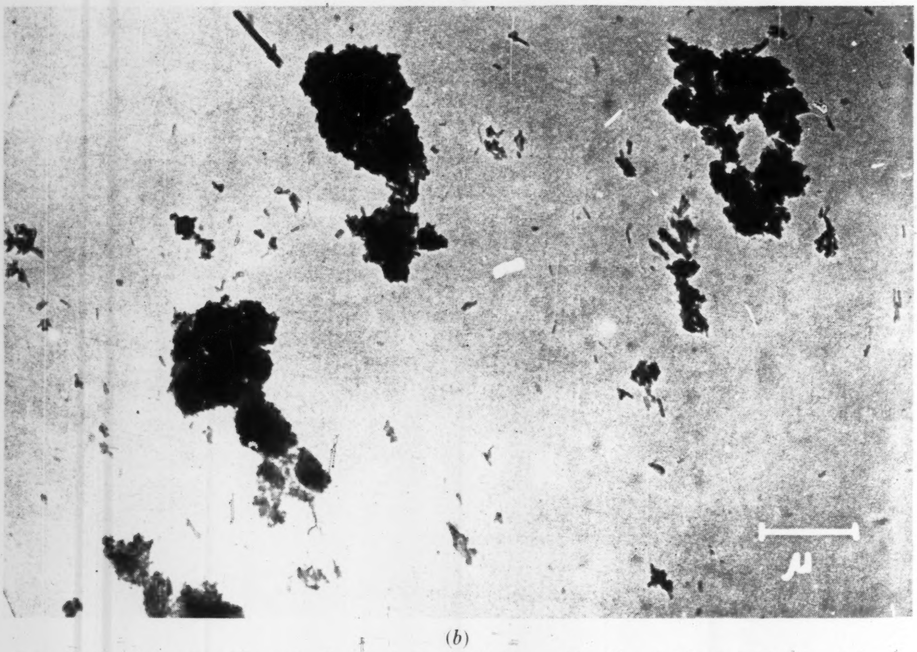
This micrograph shows the bronzed surface of an enamel incorporating iron blue as a pigment. The surface is seen to be nearly covered by a thin sheet or blanket of very fine particles loosely held together by a film of semitransparent material which is presumed to be partially degraded vehicle. These sheets of material were stripped from the surface by the replica (hence, the white appearance). The presence of iron blue pigment in these sheets of material was established by selected area electron diffraction of sheets pulled from the paint surface during replication. In the darker areas of the micrograph, which define the bulk paint surface, the degree of roughening is seen to be much less severe than was the case for the chalked surfaces examined previously. It can be seen that neither these thin, loose sheets nor the slight surface roughening is revealed adequately by the light microscope.

Figure 7(*b*) represents an electron micrograph of a dispersion of iron blue pigment in xylene. The pigment particles are very small—less than 50 mμ in diameter. This size is close to the limit of resolution of the electron microscope operating at the magnification of 15,000 × at which this picture was made. A comparison of the pigment with the tiny particles in the sheet of material of Fig. 7(*a*) shows them to be quite similar. Although it is difficult, for such small particles, to state whether a size or shape change has occurred, electron diffraction studies before and after weathering have indicated no change in chemical composition.

Another example of a typical bronzed surface is shown by an enamel containing gold paste (Fig. 8(*a*)). Again a loose blanket of pigment and degraded vehicle covers much of the paint surface. An important characteristic of a bronzed surface is that it suffers a much smaller loss in gloss than does a chalked surface.



(*a*)



(*b*)

FIG. 9(*a*).—Amberine Maroon, Bronzed Surface, Gloss 66. (*b*).—Amberine Maroon Pigment, Particle Size—less than 50; and 50 by 200 mμ.

For example, the iron blue panel retained a gloss of 44 and the gold paste panel a gloss of 59 after weathering, while the gloss of titanium dioxide fell to 24 and that of red oxide to 13. Gold paste pigment (Fig. 8(*b*)) appears very similar to iron blue. The particle size is again less than 50 m$\mu$.

The previous examples of bronzing have illustrated formulations containing inorganic pigments. An enamel containing an organic pigment, amberine maroon, also degraded to give a bronzed appearance (Fig. 9(*a*)). Like the previous examples, the vehicle displayed a characteristic fine, over-all roughening. This slight roughness suggests that tiny particles have been uncovered. Unlike the previous examples, however, it is difficult to locate discrete particles of pigment in the loose and separated sheets. In this case the pigment may have been lost from the surface or degraded in weathering. It may be significant that amberine maroon exhibits a pronounced solubility in xylene. Consequently, the pigment may have partially dissolved in the vehicle at some stage of its formulation or application.

The appearance of the original amberine maroon pigment is shown in Fig. 9(*b*). The pigment consists primarily of very small particles or agglomerates of small particles. A considerable amount of small rodlike particles is also visible. None of these rodlike particles can be seen on the surface of the degraded paint, however. The existence of most of the pigment in very small particle sizes is in agreement with the small particles observed in the other examples showing bronzing.

A definition for bronzing similar to that given above for chalking suggests itself: bronzing is the progressive shrinkage and degradation of a paint surface which manifests itself in the extensive release of sheets of pigment-containing degraded vehicle with the microstructure of the bulk surface being a function of the pigment size and shape.

It will be noted that this definition is similar to that for chalking in some respects. The mechanism of degradation is postulated as being essentially the same for the two types of failures. The surface roughness or morphology in both cases is regarded as being determined by the pigment size and shape.

Nevertheless, it is felt that there are a number of very significant differences between the two types of weathering defects. Bronzing is characterized by the release of pigment and degraded vehicle *in sheets* on the paint surface. Moreover, it has been observed that, within the limits of the pigments studied here (six chalking and nine bronzing formulations), bronzing is always associated with small pigment sizes (less than about 100 m$\mu$ in size) while chalking is always associated with pigment sizes larger than about 100 m$\mu$. Thus, the above definitions must be modified to include this distinguishing characteristic of pigment size.

Since pigment size and shape appear to be the main factors responsible for surface roughness of a weathered film, it is at once apparent that weathered bronzed surfaces should retain a higher gloss than weathered chalked surfaces. Table II gives the gloss readings for all the bronzed panels examined, and these gloss readings are seen by comparison with those of Table I to be in fact significantly higher than those for the chalked panels. The loose sheets of material, oriented as they are parallel to the enamel surface, do not significantly reduce the gloss.

In addition to determining the surface roughness of the paint in bronzing and chalking, the very small size of the pigment in bronzing may likewise be responsible for the preservation of the sheets through a reinforcing action. The reinforcement of organic materials by fillers is best understood in rubber technology, where it is well known that particle size is a controlling factor in the action of carbon black fillers. Very finely divided carbon blacks give the greatest reinforcing action in rubber hydrocarbons, an effect which is also influenced by the structure of the black. Thus bronzing may be pictured as the degradation of the surface layer of the film where the degraded layer is loosely held together by the reinforcing action of the very small pigment particles. Under the tensile force of the replication technique, these sheets of degraded material containing the pigment are pulled from the surface resulting in the typical micrographs of Figs. 7 to 9.

A typical visual effect associated with bronzing is the iridescent appearance of the surface. This effect may be the result of the fine, uniformly distributed pigment particles in the degraded sheets acting like a diffraction grating toward the incident light. Alternatively, it may be an interference phenomenon similar to that obtained with a thin film of oil on a water surface. The exact nature of the effect will require additional study, but the fact that iridescence of the bronzed film is lost after the replica is pulled off indicates that this effect is associated with the thin degraded sheets on the surface.

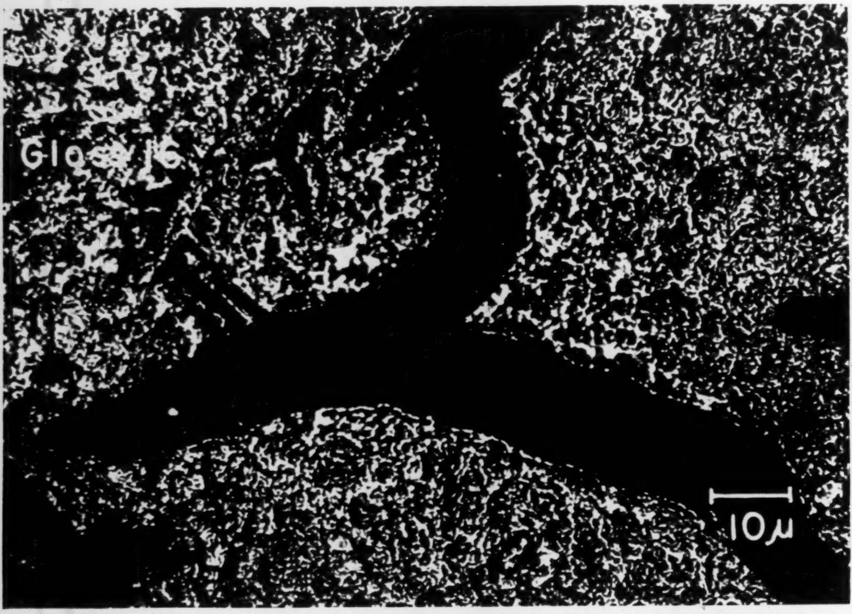


FIG. 10.—Caddy Red, Light Micrograph, Aluminized Surface, Gloss 16.

Other paints, besides those illustrated, which fail by bronzing are listed in Table II along with visual ratings of the defects. The paints are listed in the order of increasing particle size of the pigment. It will be noted that the visual ratings all include chalking as a secondary defect, although chalking in agreement with the electron microscope definition did not occur. This can be explained on the basis that the main visual test for chalking is to wipe the paint with a finger to see if color can be removed. It is felt that in severe bronzing sufficient pigment is released in the thin sheets to yield a positive result by the finger wiping test. With this consideration in mind the agreement is quite good between visual rating and microscopic appearance.

## CHECKING TYPE PIGMENTS

Visually, checking appears in a panel as a fine network of cracks or as short isolated crevices. Both types of appearance may occur either in localized areas or spread over the entire panel.

Very few of the panels examined revealed degradation by checking with little or no evidence of another type of degradation. The four panels (two of them clear vehicle) which did satisfy this criterion are listed in Table III.

In the case of severe checking, where the film contained relatively large checking cracks, lower magnification light micrographs were found to show the general condition of the surface better than electron micrographs. The typical form of "figure" or "crowsfoot" checking in which three or more crevices seem to radiate from a common point is illustrated in Fig. 10, a light micrograph of a metallized panel surface.

The large size of crevices makes replication for electron microscopy difficult. This large size likewise frustrates attempts to show the extent of checking with the electron microscope, which is best suited to studies at higher magnifications. The light micrograph of Fig. 11, at a magnification of 200 ×, indicates the extent of film destruction caused by checking in a clear film. Included in the micrograph are areas from which the vehicle has completely disappeared.

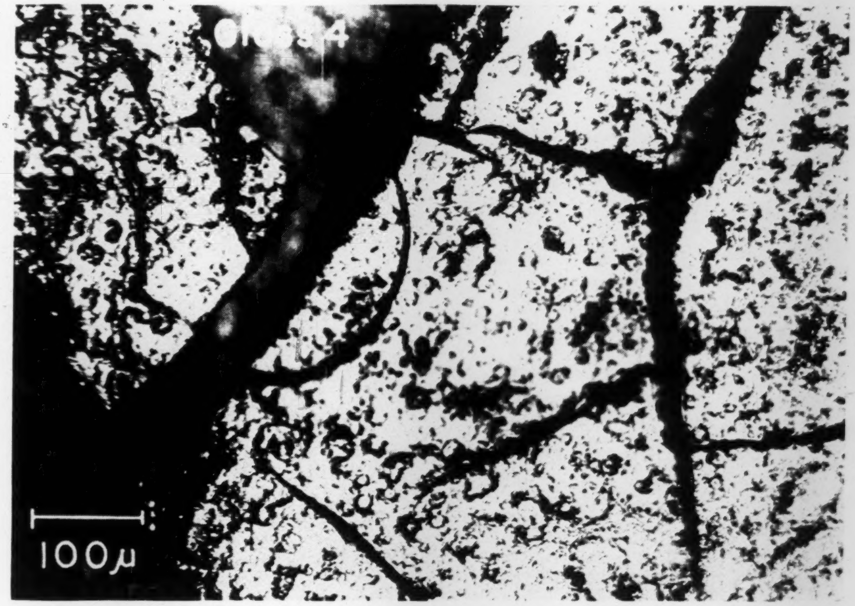


FIG. 11.—Clear Vehicle Film, Light Micrograph, Aluminized Surface.

The appearance of a checked surface has been established by previous electron microscope work (16) as consisting of large crevices with a V-shaped cross-section which penetrate deeply into the paint film. As shown by Fig. 12, this defect occurs readily in an unpigmented film. Thus checking establishes itself as the primary mode of weathering of clear vehicle. Some crevices on this panel, as seen in light micrographs, extend completely through the vehicle film to the substrate surface. In the electron microscope many areas on this panel show only rust and a coarse crystal structure resembling a phosphate coating indicating the complete loss of the vehicle film. Some portions of the vehicle surface have developed a flaky appearance due to erosion of degraded vehicle. During this weathering process gloss has dropped to a reading of 4 on the Gardner glossmeter. A similar panel of clear vehicle was exposed in Detroit. In the milder atmosphere of Detroit, weathering conditions are much less severe than for an equal exposure period in Florida. As indicated in Table III, the gloss of this panel dropped only to 64, but the mode of degradation was identical to that of the Florida exposed panel.

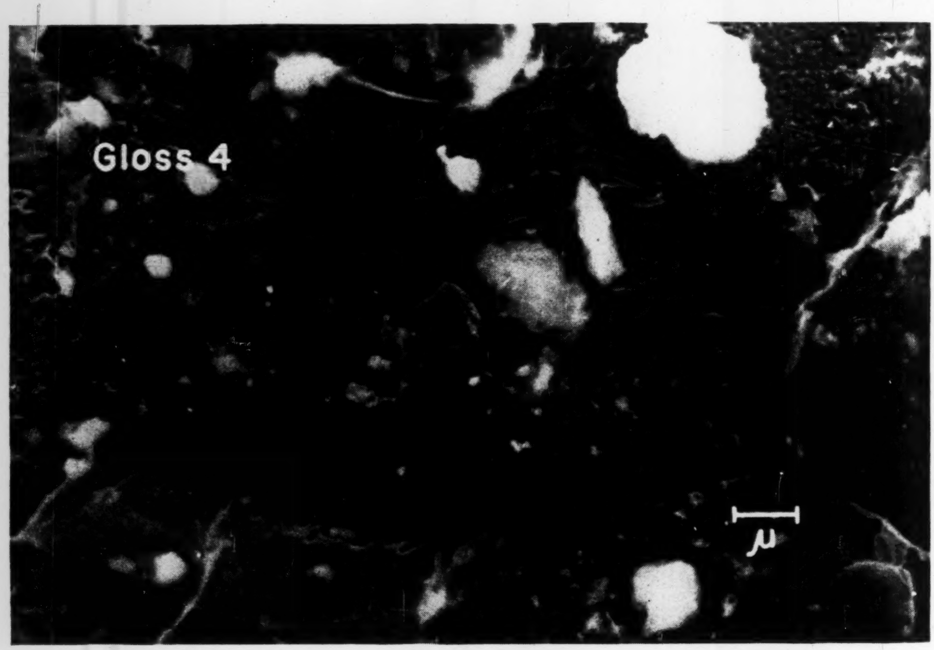
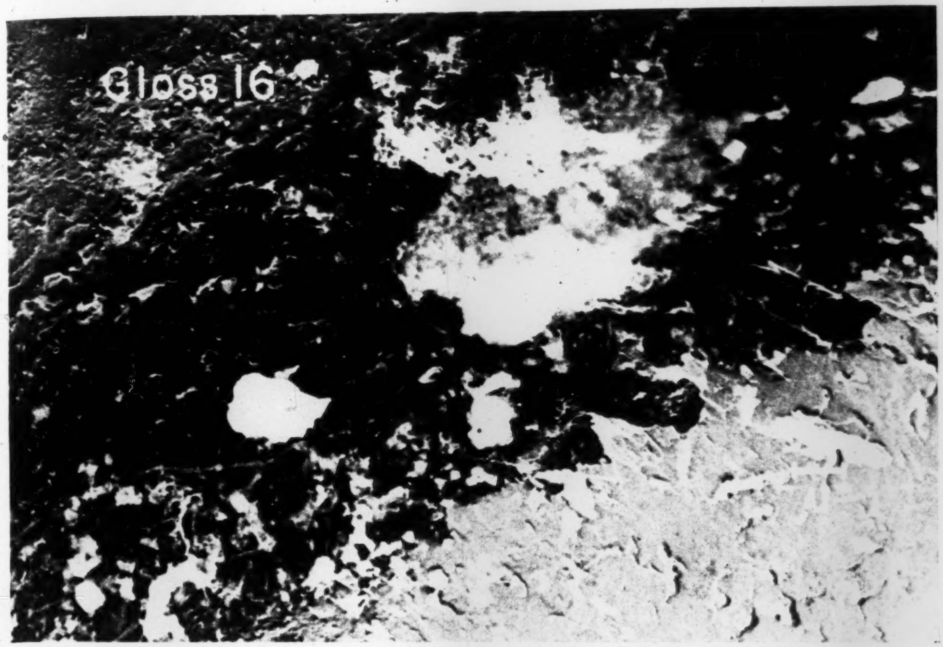


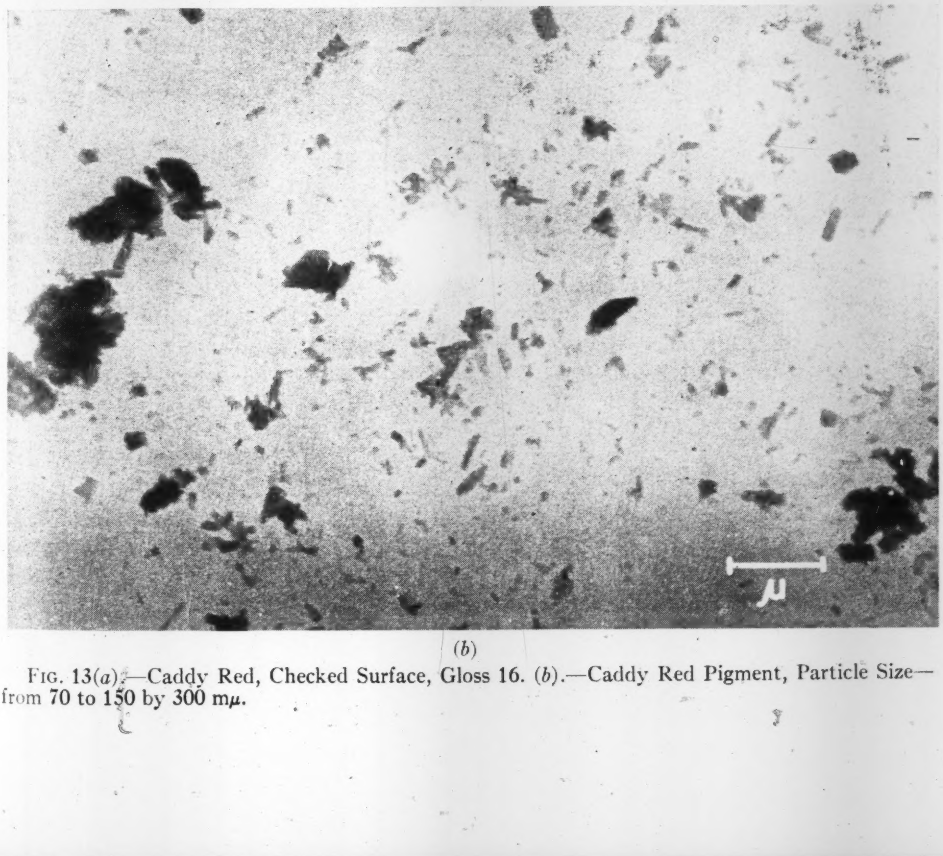
Fig. 12.—Clear Vehicle, Checked Surface, Gloss 4.

Checking is regarded as a weathering defect resulting from the degradation of vehicle to a substantial depth of the total paint film. The fact that a checked appearance is characteristic of weathered clear vehicle suggests that its presence to a substantial degree in pigmented films is evidence of the inability of the pigment to protect underlying layers of vehicle from harmful incident radiation. Alternatively, or acting together with this deficiency, the pigment may be present in such a size or concentration that it exerts insufficient reinforcing action in the bulk film. The particular pigment characteristics conducive to the propagation of checking will require considerable further study.

Caddy red is a good example of a pigment which, when incorporated into an



(a)



(b)

FIG. 13(a).—Caddy Red, Checked Surface, Gloss 16. (b).—Caddy Red Pigment, Particle Size—from 70 to 150 by 300 mμ.

enamel, permits extensive checking. An electron micrograph of caddy red is shown in Fig. 13(*a*). Extending across the micrograph is a typical crevice indicating vehicle shrinkage; also a few distinctive pigment particles can be seen. A slight erosion of the surface can be observed, and this together with the extensive checking accounts for the almost complete loss of gloss characteristic of checking. Caddy red pigment is less regular in either size or shape than many of the pigments included in this study. Figure 13(*b*) indicates the characteristic appearance to be flakes of widely varying size.

### COMBINATION TYPE PIGMENTS

The most common degradation observed involved two or more principal types of defects. Although several cases were found of both chalking and bronzing according to microscopic appearances, the more common was bronzing and checking. Table IV lists these panels beginning with chalking and bronzing and proceeding to bronzing and checking.

In the light microscope the combination of chalking and checking is easily identified as seen in Fig. 14, while a combination of bronzing and checking usually looks like checking alone because of the low contrast provided by the loose sheets of pigment. Similarly, a combination of chalking and bronzing is difficult to identify with the light microscope because the available contrast gives the appearance only of mild chalking.

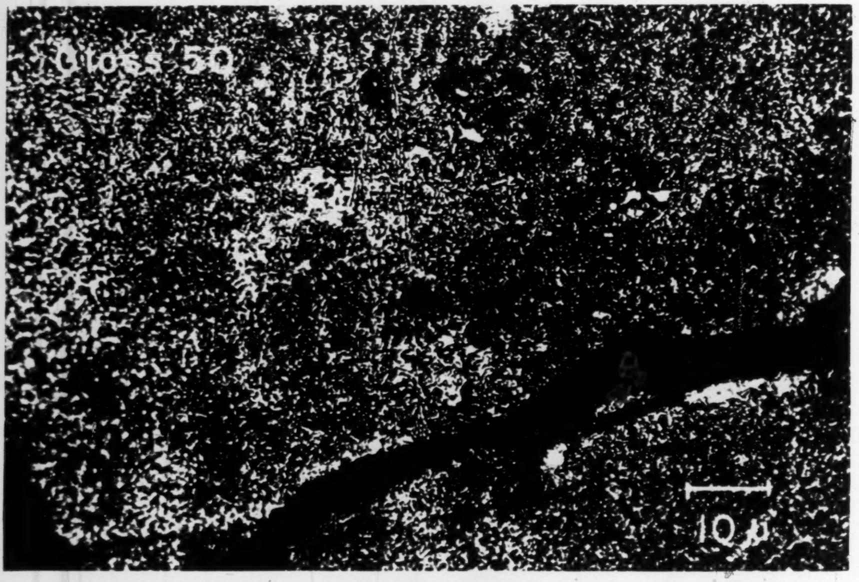
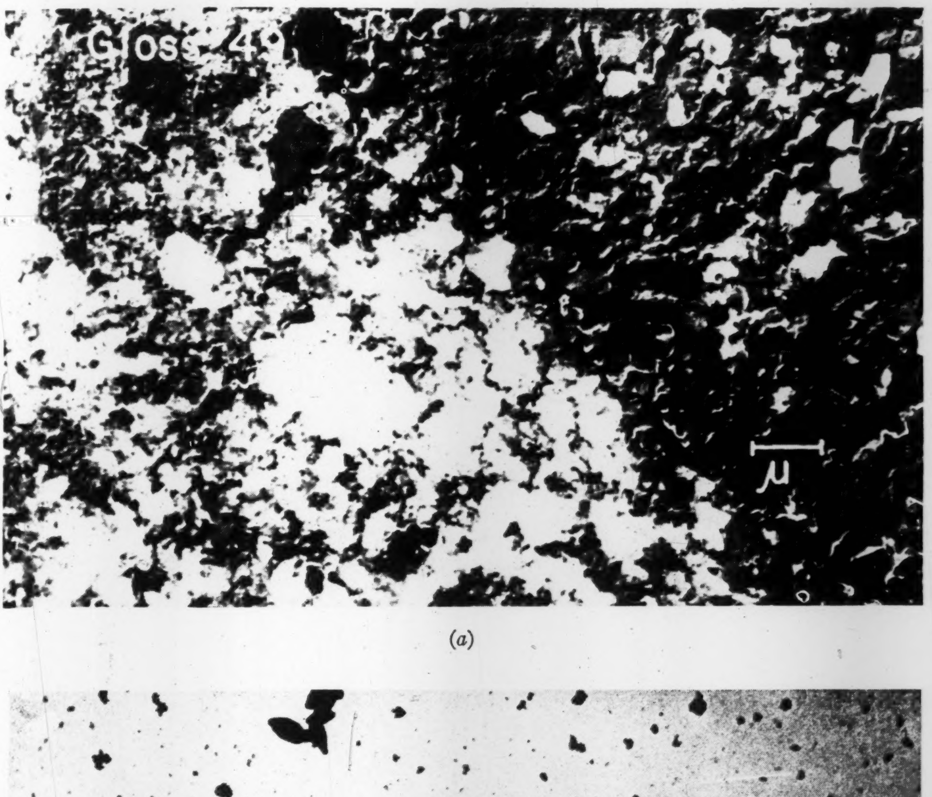


FIG. 14.—Molybdate Orange, Light Micrograph, Aluminized Surface, Gloss 50.

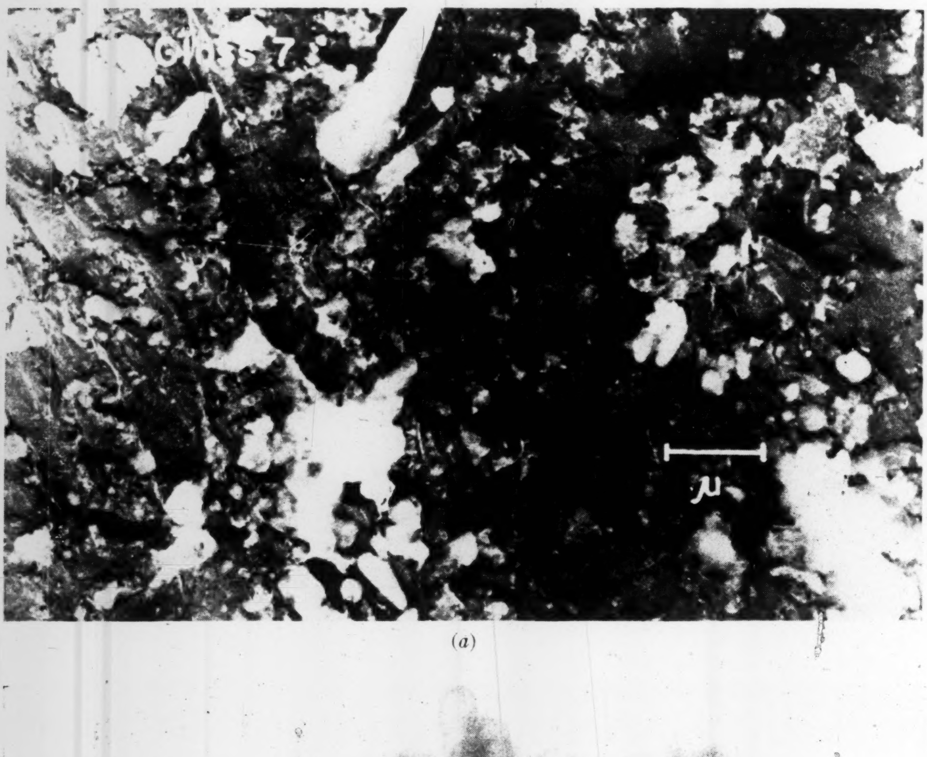
An example of both chalking and bronzing is shown by an electron micrograph of chrome yellow (Fig. 15(*a*)) in which single particles of medium and large sized pigment resemble chalking, although sheets of material like those characteristic of bronzed surfaces are also present. A micrograph of the pigment (Fig. 15(*b*)) indicates that it is composed of both large diamond-shaped particles and very small particles. This dual size range is consistent with the appearance



(*a*)

(*b*)

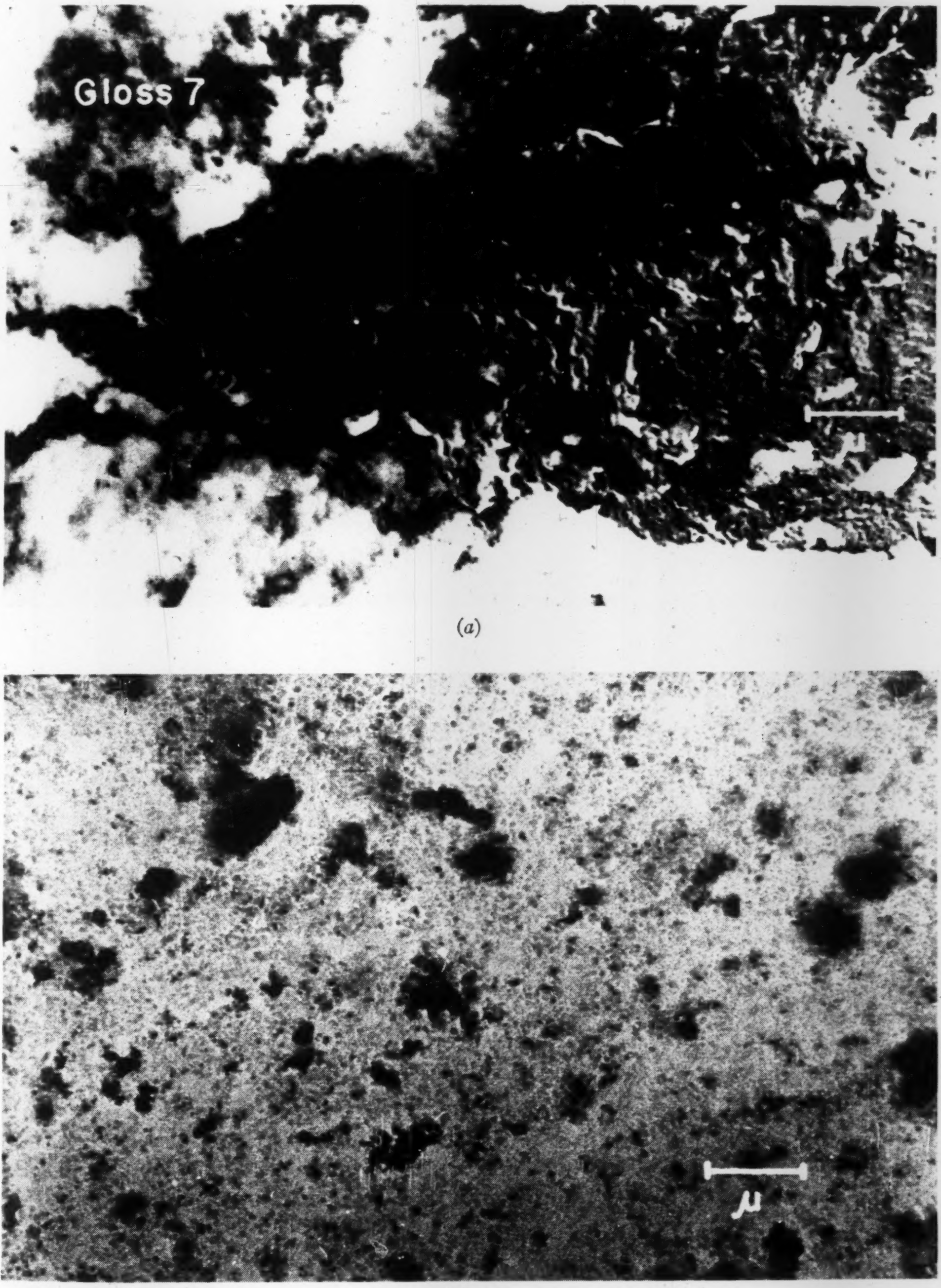
FIG. 15(*a*).—Chrome Yellow, Chalking and Bronzing, Gloss 49. (*b*).—Chrome Yellow Pigment, Particle Size—less than 50 m$\mu$; 300 m$\mu$.



(a)

(b)

FIG. 16(a).—Toluidine Red, Chalking and Bronzing, Gloss 7. (b).—Toluidine Red Pigment, Particle Size—above 1 mμ.



(*a*)

(*b*)

Fig. 17(*a*).—Chrome Hydrate Green, Bronzing and Checking, Gloss 7. (*b*).—Chrome Hydrate Green Pigment, Particle Size—less than 50 mμ.

of both bronzing and chalking seen on the degraded film. Although chrome yellow is classified as a single pigment, it consists of two inorganic salts, lead chromate and lead sulfate. However, electron diffraction of each of the two fractions after separation in a centrifuge showed only the presence of lead chromate.

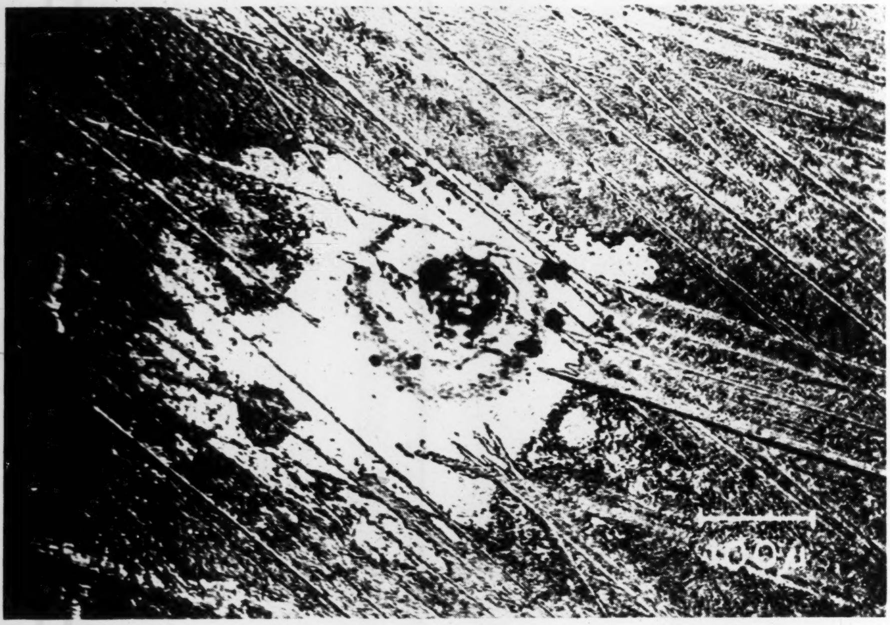


FIG. 18.—Iron Blue Showing Bronze Spot, Light Micrograph, not Aluminized.

An example of a paint which shows chalking, bronzing, and occasional checking is found in toluidine red (Fig. 16(*a*)). The general surface roughness seems somewhat greater than that typical of bronzing, and this factor, together with the presence of relatively large individual, rectangular particles, would indicate a chalk rating. Nevertheless, numerous small areas of fine particles and sheets of material are scattered about the surface indicating a definite bronzing tendency. A gloss of 66 is retained by the weathered panel, indicating that the surface roughness shown by this particular micrograph may be deceptively high.

The pigment (Fig. 16(*b*)) shows remarkably large particles, only the smallest of which were found on the degraded surface. This appearance suggests that either the large particles were washed away or broken down into smaller particles. Since no small particles of bronzing size were found in the pigment dispersion, the latter condition is more likely. The pigment tended to melt in the heat of the electron beam as indicated by the small bubbles appearing in some particles and the fusing together of other particles (Fig. 16(*b*)). The additional possibility exists that the pigment may exist partially in solution in the vehicle and recrystallize on the surface in a smaller particle size during weathering.

The most common combination effect found in the group of panels was bronzing and checking. In our previous work it was thought that organic pigments may give less protection to the underlying vehicle than inorganic pigments. However, in low concentrations, an inorganic pigment may permit checking. Chrome hydrate green (Fig. 17(*a*)), which is inorganic, shows a surface broken up by checks. Over the film between the crevices are found sheets of bronzing material. As seen in Fig. 17(*b*), the pigment consists of very fine particles which are typical of a bronzing pigment. The severe loss of gloss by the panels is due to the extensive checking which it suffered. Checking seems to occur much more readily in conjunction with bronzing than with chalking. The significance of the association of these defects lies in the implication that bronzing pigments provide inadequate protection from weathering agents capable of causing bulk film breakdown. Other formulations which fail through bronzing and checking are listed in Table IV.

A particular type of degradation not easily characterized under the previous headings is bronze spot. These spots can be seen visually on bronzed panels and appear to be limited areas of intense bronzing usually accompanied by considerable iridescence. As seen in the light micrograph (Fig. 18), a typical bronze spot appears as an iridescent sheet surrounding a central spot of rust or a pit.

Studies of bronze spot can be conducted better with the light microscope than with the electron microscope, since the field of view with the latter instrument is too restricted to include the entire area of the defect. However, a series of electron micrographs taken along a radius from the center of the spot showed the central spot to be a badly torn up area of severe degradation. A loose sheet of material extends from this central area, growing thinner or less dense as the distance from the center increases. As shown in the center of Fig. 2, a light micrograph of a titanium dioxide pigmented panel, chalking films also reveal pits and rust spots surrounded by areas of different appearance from the general surface.

Similarly, examination of areas of degraded, unpigmented vehicle indicated bronze spots and included the iridescent sheets in many places. This information suggests that the phenomenon of bronze spot is not peculiar to bronzing surfaces, nor does its formation appear to require the presence of pigment in the film. The origin of the iridescent sheet and an indication of whether the central spot was initiated at the film-air or the film-subsurface interface could not be determined by this study. It is probable, however, that the so-called bronze spot may be due to a pore or hole in the original film which permits attack at the metal interface by corrosive agents.

## CONCLUSIONS

From the microscopical standpoint, chalking consists of the progressive shrinkage and erosion of a paint film surface resulting in the exposure and release of discrete pigment particles and small groups of particles. The effect creates a surface whose roughness is related to the pigment size, and whose morphology is determined by the effective pigment size and shape. Similarly, bronzing is the progressive shrinkage and degradation of a paint surface which manifests itself in the extensive release of sheets of material consisting of pigment and partially degraded vehicle. The microstructure of the bulk surface is a direct function of pigment size and shape.

In the absence of checking, when the pigment particle size in an enamel is greater than about 100 m$\mu$, the enamel will chalk. When the pigment particle size is less than about 100 m$\mu$, the paint will bronze. When the pigment is present in a formulation in size ranges less than 100 m$\mu$ and also greater than 100 m$\mu$, both bronzing and chalking will occur. Also, since the loss of gloss is less for bronzing than for chalking films, the gloss (and hence the surface roughness) appears to be at least qualitatively related to pigment size.

Checking is regarded as a weathering defect resulting from the degradation of vehicle to a substantial depth of the total paint film. It is the primary mode of degradation of unpigmented vehicle. It occurs also in pigmented films, sometimes as the principal defect but more often in conjunction with another type of defect. In a majority of these latter cases checking is associated with bronzing, but rarely it is also found with chalking pigments. Checking is believed to be evidence of the inability of the pigment to protect underlying layers of vehicle from damaging incident radiation.

There appears to be no strict relationship between chemical composition of the pigment and the type of defect associated with it. The list of chalking pigments includes both inorganic and metallo-organic pigments. These two types, as well as organic pigments, are also associated with bronzing and checking (see Tables II and III). From the results of a limited amount of electron diffraction study, it is felt that the examples of paint degradation included in this study do not involve a change in the chemical composition of the pigments, although the less stable pigments (for example, toluidine red) may change in physical character. Considerable additional work must be done, however, before a positive conclusion can be made in this regard.

Some pigments (chrome yellow, chrome green, chrome orange, molybdate orange, indo blue and indo orange) which are regarded by paint manufacturers as being single pigments actually consist of two or more distinct chemical compounds and exhibit dual size distributions. All except chrome green failed by some combination of bronzing, chalking, or checking. Chrome green bronzed only (see Tables II and IV).

Bronze spot appears to be a localized phenomenon. Observations of a similar attack with both chalking paints and clear vehicle indicate it is a defect caused by a local weakness in the film rather than by a particular pigment characteristic. The more obvious appearance of bronze spot on bronzed surfaces than on chalked surfaces appears to result from its enhancement of the usual iridescence found on bronzed films.

## FUTURE WORK

The results of the present work indicate that much further study is needed before the role of pigment in paint weathering can be fully understood. Independent verification is being made of the role of pigment size in determining bronzing or chalking without the possible complicating interference of other factors such as varying light absorption by the pigment and pigment shape or concentration. To accomplish this, films are being formulated which contain the same pigment at the same volume concentration but in a different pigment size range. These films will be weathered naturally and artificially. Carbon black is suitable for such an investigation, since it is readily available in a wide range of sizes, some much smaller than the critical value of 100 m$\mu$ and others, such as Thermax, much larger than 100 m$\mu$. Care must be exercised to avoid those with a high structure index which would result in a much higher effective particle size due to agglomeration of particles in the vehicle.

Anatase titanium dioxide has also been obtained in a range of particle sizes, above and below 100 m$\mu$, and will be used to establish the importance of pigment size in bronzing and chalking. Using a single pigment composition, data can also be obtained on the effect of pigment concentration on the type and severity of defects occurring in weathered paint films. This is also being pursued at the present time in our laboratories.

The behavior of pigments toward incident radiation seems to be quite important in weathering. The fact that checking may occur extensively in a pigmented film suggests that studies of light absorption, scattering, and transmission by the pigment are highly desirable. As indicated by Table IV, checking seems to occur much more readily in conjunction with bronzing than with chalking. Therefore, studies are being made using visible and ultraviolet light absorption and scattering techniques in an attempt to clarify some of the significant factors involved in checking.

Other work being undertaken includes a more intensive study of so-called bronze spot to determine if it is a weathering defect or if, as seems more likely, it is a defect which occurs during the application of the paint film to the panel. The tendency of some pigments to change size on weathering and to appear differently on a weathered surface than when examined individually also merits additional study. Toluidine red may be an example of such a pigment. Kienle and Maresh (12) have noticed a recrystallization effect with toluidine toner. In the case studied by them a yellow bronze color was produced by recrystallization of the pigment to form larger particles on the paint surface during aging. Subsurface studies of paint films have been initiated in our laboratories in this connection. Diffraction studies are also being enlarged to determine whether or not changes in pigment composition occur during the course of weathering.

### *Acknowledgment:*

Recognition has been given in the body of the paper for the assistance of the Ditzler Color Division of Pittsburgh Plate Glass Co. in supplying the weathered panels and uncompounded single pigments used in this study. Mention should be made of the helpful discussions during the course of this work with Mr. R. I. Peters of Ditzler Color.

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## REFERENCES

(1) E. B. Fitzgerald, "Photooxidative Degradation of Alkyd Films," ASTM BULLETIN, No. 207, July, 1955, p. 65 (TP 137).

(2) S. Crecelius, R. Kagarise, and A. Alexander, "Drying Oil Oxidation Mechanism, Film Formation and Degradation," *Industrial and Engineering Chemistry*, Vol. 47, Aug., 1955, p. 1643.

(3) C. D. Miller, "Kinetics and Mechanism of Alkyd Photooxidation," *Preprint*, Am. Chemical Soc., *Division of Plastics, Paint, and Printing Ink Chemistry*, Vol. 16, No. 1, April, 1956, p. 31.

(4) S. Gusman and J. D. Stroupe, "Deterioration of Synthetic Enamels—Absorption of Solar Energy," *Paper No. 17*, Am. Chemical Soc., Division of Paint, *Plastics and Printing Ink Chemistry*, Vol. 15, April, 1955, p. 182.

(5) N. J. Marshall, "The Effect of Light of Different Wavelengths on the Degradation of Clear Coatings," *Official Digest*, Federation of Paint and Varnish Production Clubs, Vol. 29, Aug., 1957, p. 792.

(6) A. C. Elm, "Reevaluation of the Function of Pigments in Paints," *Official Digest*, Federation of Paint and Varnish Production Clubs, Vol. 29, April, 1957, p. 351.

(7) L. H. Wartman, "Heat and Light Degradation of Vinyl Chloride Resin," *Industrial*

*and Engineering Chemistry*, Vol. 47, May, 1955, p. 1013.

(8) S. Gusman and R. Spell, "Deterioration of Synthetic Enamels—II, Photodegradation of Benzoguanamine Resin Systems," *Preprint*, Am. Chemical Soc., *Division of Plastics, Paint and Printing Ink Chemistry*, Vol. 16, No. 2, Sept., 1956, p. 74.

(9) M. Kronstein, "Studies of the Relation Between the Structure of Pigmented Films and Their Behavior under Ultraviolet Radiation," Am. Chemical Soc., *Division of Plastics, Paint and Printing Ink Chemistry*, Vol. 15, No. 2, Sept., 1955, p. 77.

(10) D. L. Tilleard and M. S. C. Birbeck, "The Surface of Paint Films—Electron Microscopy—I," *Research Memorandum No. 141*, Paint Research Station, March, 1947.

(11) D. L. Tilleard and M. S. C. Birbeck, "The Surface of Paint Films—Electron Microscopy—II," *Research Memorandum No. 145*, Paint Research Station, Sept., 1947.

(12) R. H. Kienle and C. Maresh, "Observations on Optical Properties of Pigmented Films," *Official Digest*, Federation of Paint and Varnish Production Clubs, Vol. 26, Jan., 1954, p. 5–28.

(13) N. D. P. Smith, "The Electron Microscope in the Study of Paints," *Transactions*, Inst. of Metal Finishing, Advance Copy No. 12 (1955).

(14) S. B. Twiss, D. M. Teague, and W. L. Weeks, "Electron Microscope Study of Paint Surfaces," *Official Digest*, Federation of Paint and Varnish Production Clubs, Vol. 28, Feb., 1956, p. 93.

(15) M. M. Gerson, "Mechanism of Paint Film Breakdown—I," *Official Digest*, Federation of Paint and Varnish Production Clubs, Vol. 26, Nov., 1954, p. 1076.

(16) S. B. Twiss, D. M. Teague, and W. L. Weeks, "Electron Microscope Study of Accelerated and Natural Weathering," *Official Digest*, Federation of Paint and Varnish Production Clubs, Vol. 30, Jan., 1958, p. 7–36.

# SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SPECTROCHEMICAL ANALYSIS FOR TRACE ELEMENTS

The papers and discussions in this Symposium were presented at the Eleventh and Fourteenth Sessions of the Sixtieth Annual Meeting of the American Society for Testing Materials, held in Atlantic City, N. J., June 18, 1957. It was sponsored by Committee E-2 on Emission Spectroscopy, with D. L. Timma acting as Symposium Chairman and presiding over the sessions.

The papers included in the Symposium are:

Emission Spectrometric Determination of Oxygen in Metals—V. A. Fassel, W. A. Gordon and R. W. Tabeling
Spectrographic Determination of Trace Elements in Metals—J. A. Norris
Trace Analysis by Means of the Graphite Spark—J. M. Morris and F. X. Pink
Medical and Biological Materials—B. Vallee
Plants and Soils—W. G. Schrenk
Geological Materials—K. J. Murata

These papers with discussions were issued as *ASTM Special Technical Publication No. 221*, entitled "Symposium on Spectrochemical Analysis for Trace Elements."

# SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS—VOLUME 2

This symposium is the second of a series which will be sponsored jointly by ASTM and the Atomic Industrial Forum. The first symposium was presented at the Second Pacific Area National Meeting of the Society, Los Angeles, Calif., September 20, 1956, and the papers were subsequently published in the Symposium on Radiation Effects on Materials—Volume 1, STP No. 208.

The papers and discussions in this Symposium were presented at the Twenty-fourth and Twenty-sixth Sessions of the Sixtieth Annual Meeting of the American Society for Testing Materials, held in Atlantic City, N. J., June 20, 1957. It was sponsored jointly by Committee E-10 on Radioisotopes and Radiation Effects and the Atomic Industrial Forum with C. C. Woolsey acting as Symposium Chairman and Messrs. Woolsey and G. D. Calkins presiding over the session.

The papers included in the Symposium are:

Selection of Structural Materials for Components of Nuclear Power Plants—W. L. Fleischmann

Problems in Standardization of Techniques in Radiation Studies—M. J. Feldman and R. H. Fillnow

Application of the Battelle Research Reactor to Radiation Effects Studies—J. W. Chastain

A Technique for Measurement of Electrical Resistivity of Radioactive Metals—C. L. Boyd

Techniques of Tension Testing Irradiated Materials at Elevated Temperatures at Hanford—W. S. Kelly, D. C. Kaulitz, and R. E. Hueschen

Techniques for Making Visual Examinations and Dimensional Measurements at Hanford's Radiometallurgy Laboratory—D. L. Zimmerman

Hanford's Improved Remote Metallographic Equipment—J. R. Morgan

Effect of Heat Treatment and Burnup on Radiation Stability of 10 per cent by Weight Molybdenum-Uranium Fuel Alloys—G. D. Calkins, D. O. Leeser, J. E. Gates, F. A. Rough and A. DelGrosso

Engineering Effects of Radiation on Nuclear Fuels—B. Lustman

A Survey of the Radiation Stability of Hydrocarbon Fuels—J. B. Carroll, R. O. Bolt, and J. A. Bert

Selection of Organic Materials as Reactor Coolant-Moderators—E. L. Colichman and R. H. J. Gercke

Effects of Radiation on Electronic Components—I. Semiconductor Devices—M. A. Xavier, G. J. Rotariu, S. Nelson, A. Yefsky, and A. Walters

These papers with discussion were issued as *ASTM Special Technical Publication No. 220*, entitled "Symposium on Radiation Effects on Materials—Vol. 2."

# SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON DETERMINATION OF DISSOLVED OXYGEN IN WATER

The papers and discussions in this Symposium were presented at the Twenty-eighth Session of the Sixtieth Annual Meeting of the American Society for Testing Materials, held in Atlantic City, N. J., June 20, 1957. It was sponsored by Committee D-19 on Dissolved Oxygen in Water, with R. C. Adams acting as Symposium chairman and F. U. Neat presiding over the session.

The papers included in the Symposium are:

A Study of the Accuracy of Methods of Testing for Dissolved Oxygen in High Purity Water—K. G. Stoffer

Polarographic Measurement of Dissolved Oxygen—W. W. Eckenfelder, Jr. and Conrad T. Burris

The Beckman Dissolved Oxygen Analyzer—Thomas Finnegan and Ross C. Tucker

Evaluation of Hartmann and Braun Dissolved Oxygen Recorder for Boiler Feedwater—A. J. Ristaino and A. A. Dominick

Determination of Dissolved Oxygen by Means of a Cambridge Analyzer—H. A. Grabowski

These papers with discussion were issued as *ASTM Special Technical Publication No. 219*, entitled "Symposium on Determination of Dissolved Oxygen in Water."

# SYMPOSIUM ON WAX

A Symposium on Wax was sponsored by Technical Committee M on Petroleum Wax of Committee D-2 on Petroleum Products and Lubricants. Technical Committee M is a joint activity of ASTM and the Technical Association of the Pulp and Paper Industry (TAPPI). The papers in this publication deal with the correlation of functional qualities of wax with composition and physical properties. The Symposium was held on February 19, 1958, in New York City, in conjunction with the Annual Meeting of TAPPI.

The Symposium consists of the following papers:

Sealing Strength of Wax-Polyethylene Blends—D. S. Brown, W. R. Turner, and A. C. Smith, Jr.

The Relationship of Wax Crystal Structure to the Water Vapor Transmission Rate of Wax Films—R. C. Fox

Composition *versus* Properties of Microcrystalline Waxes—W. P. Ridenour, I. J. Spilners, and P. R. Templin

Solid Petroleum Hydrocarbons and Their Effect on Wax Properties—R. T. Edwards

Relation Between Composition and Blocking Temperature of Paraffin Waxes—K. G. Arabian

Effect of Refining Conditions on Wax Quality—R. L. Murphey and W. B. Chandler, Jr.

Gloss and Gloss Stability of Waxed Papers—F. J. Hughes and D. C. Walker

The Effect of Paper-Making Materials on Wax Oxidation Rates—G. G. Rumberger

Factors Influencing the Staining Tendency of Wax—Joseph Phillips

The papers will appear in *TAPPI*. Reprints may be secured from ASTM Headquarters.

# SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON NONDESTRUCTIVE TESTS IN THE FIELD OF NUCLEAR ENERGY

This symposium evolved from the meetings of an informal committee within the Atomic Energy Commission contractors dealing with the general subject of nondestructive testing. As the information that was developed in the committee meetings was in the process of being declassified, there was a desire for a national meeting where papers on the subject could be presented.

The symposium was held in Chicago, Ill., on April 16–18, 1957, cosponsored by the American Institute of Chemical Engineers, Nuclear Division; American Nuclear Society; American Society for Testing Materials; Atomic Industrial Forum; and Society for Nondestructive Testing. The executive committee of the symposium was composed of W. J. McGonnagle, Argonne National Laboratory, Lemont, Ill., Chairman; S. Lawroski, Argonne National Laboratory, Lemont, Ill.; F. L. LaQue, The International Nickel Co., New York, N. Y.; Stuart McLain, Argonne National Laboratory, Lemont, Ill.; H. Migel, Magnaflux Corp., Chicago, Ill.; J. L. Swanson, General Electric Co., Cincinnati, Ohio; and S. A. Wenk, University of California, Radiation Laboratory, Livermore, Calif. Two evening discussion panel sessions were held as a special feature of the meeting. Prepared questions were submitted to a panel which included the authors and speakers for the day. A number of papers were presented by title only at the symposium but have been included in the symposium proceedings.

The Symposium includes the following papers:

- Introduction—W. J. McGonnagle
- Nondestructive Testing in the Nuclear Energy Field—Stuart McLain
- Introduction to Eddy Current Methods and Techniques—H. L. Libby
- Survey of Ultrasonic Methods and Techniques—S. A. Wenk
- Survey of Radiation Techniques—G. H. Tenney
- Production Inspection of Pipe and Tubing by the Immersed Ultrasonic Method—R. B. Oliver, R. W. McClung and J. K. White
- An Eddy Current Test for Capillary Tubing—W. R. Plant and C. Mannal
- Inspection of Small Diameter Tubing by Eddy Current Methods—J. W. Allen and R. B. Oliver
- A Nondestructive Test for Intergranular Corrosion in Stainless Steel—R. C. Robinson
- Two Applications of Eddy Current Instruments to Testing of Zircaloy Core Components—R. A. Betz
- The Use of Penetrants for Inspection of Small Diameter Tubing—R. B. Oliver, G. M. Tolson, and A. Taboada
- An Approach to Versatility in Eddy Current Testing—P. D. Edwards
- Radiography of Materials Used in the Nuclear Energy Field—J. W. Dutli and D. E. Grimm
- Radiography with Thulium Sources—S. S. Sidhu, R. P. Campos, and D. D. Zauberis

Measurement of Density of Uranium Metal by Radiation Absorption—J. P. Scheuer and P. R. Morris

Ultrasonic Testing as a Method of Determining Variables in Processing Zircaloy and Hafnium—Edwin W. Fink

Minimizing the Effect of Probe-to-Metal Spacing in Eddy Current Testing—C. J. Renken, Jr., and D. L. Waidelich

Reduction of Probe-Spacing Effect in Pulsed Eddy Current Testing—D. L. Waidelich

Eddy Current Measurement of Clad Thickness—J. W. Allen, R. A. Nance, and R. B. Oliver

Testing of Cylindrical Fuel Elements Using the Cyclograph—W. J. McGonnagle

Nickel Thickness Gage—L. H. Cook

A High Precision Density Times Thickness Gage—G. M. Taylor

Ultrasonic Transmission Tester for Detection of Unbonded Areas—J. D. Ross and R. W. Leep

An Ultrasonic Scanner and Recording System—W. N. Beck

Applications of Lamb Waves in Ultrasonic Testing—D. C. Worlton

Radiography of Fuel Elements and Fuel Materials Using Cesium-137—Merle L. Rhoten

Eddy Current Techniques for Testing Liquid Metal Bonding—C. J. Renken, Jr., and W. J. McGonnagle

Electrode Potential Method of Bond Testing—W. G. Marburger, J. H. Monaweck, and W. J. McGonnagle

Nondestructive Bond Inspection Test by Electric Resistance Measurement for Complete Flat-Plate Fuel Element Subassemblies—C. V. Weaver, W. H. Goldthwaite, S. L. Fawcett, and R. W. Dayton

Measurement of Cladding Thickness on Uranium by Autoradiography—G. E. Bradley, W. J. McGonnagle, and P. R. Gonzales

Radiographic Inspection of Nuclear Core Materials and Components—A. E. Oakes

Techniques for X-Ray Examinations of End-Weld Closures of Cylindrical Fuel Elements—S. S. Sidhu

Helium Leak Detection Techniques—W. H. Pappin

Helium Leak Detector Test for Nuclear Reactor Welds—A. H. Barnes, F. A. Smith, and E. A. Wimunc

A Nickel Depth Meter—G. D. Linsey and H. L. Libby

Thermal Testing of Reactor Fuel Elements—J. H. Monaweck and W. J. McGonnagle

A Recording X-ray Photometer—W. R. Plant

Two Methods for Measuring Total Uranium Content in Co-Extruded $AlU_3O_8$ Reactor Fuel Plates—G. E. Bradley, E. G. Leverenz, and W. J. McGonnagle

Approximate Determination of Enrichment of Reactor Fuel Element Plates with Improvised Gamma Ray Spectrometer—G. E. Bradley

A Nondestructive Method for Fuel Assaying—S. G. Forbes

A Technique for Gamma Radiographic Inspection of Parallel-Plate Fuel Assemblies—G. D. Calkins, M. Podereskin, R. B. Price, and N. M. Ewbank, Jr.

A Panoramic Camera for Inspecting Walls of Deep Narrow Slots—G. G. Cocks and C. M. Schwartz

These papers with discussion were issued as ASTM *Special Technical Publication No. 223* entitled "Symposium on Nondestructive Tests in the Field of Nuclear Energy."

# SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON INSULATING OILS

This Symposium on Insulating Oils was sponsored by Subcommittee IV on Liquid Insulation of ASTM Committee D-9 on Electrical Insulating Materials and presented during the meetings of Committee D-9 in Roanoke, Va., February 26, 1957. It was the seventh session in a series of symposia on insulating oils, presented under the sponsorship of Subcommittee IV over a period of about eleven years.

The first two symposia were published in ASTM BULLETINS No. 146, May, 1947, and No. 149, December, 1947. The proceedings of the third, fourth, fifth, and sixth symposia were printed as *ASTM Special Technical Publications Nos. 95, 135, 152,* and *172,* respectively.

The Symposium includes the following papers:

Introduction—L. B. Schofield
Evaluation of Laboratory Tests as Indicators of the Service Life of Uninhibited Electrical Insulating Oils—T. A. McConnell
The Use of Solid and Liquid Catalysts for the Accelerated Aging Testing of Transformer Oil—Edward L. Raab

These papers with general discussion were issued as *ASTM Special Technical Publication No. 218* entitled "Symposium on Insulating Oils."

# SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON COMPOSITION OF PETROLEUM OILS

This Symposium was arranged by Research Division IV of ASTM Committee D-2 on Petroleum Products and Lubricants under the sponsorship of the Division of Refining of the American Petroleum Institute, through a Symposium Committee of which R. A. Klett, Universal Oil Products Co., served as chairman.

These papers were presented in New Orleans, La., on February 8–9, 1957, at four sessions—one of the four sessions being devoted entirely to open discussion. The first session was presided over by Mr. W. E. Scovill, The Standard Oil Co. (Ohio), Cleveland, Ohio; the second session was presided over by Mr. Eugene J. Rosenbaum, Sun Oil Co., Norwood, Pa.; the third session was presided over by Mr. Robert L. LeTourneau, California Research Corp., Richmond, Calif.; the fourth session was presided over by Mr. Harold M. Smith, U. S. Bureau of Mines, Bartlesville, Okla.

The papers presented were:

Preface—Harold M. Smith
Introduction—R. A. Klett

### Separation

Introduction—W. E. Scovill
Summary of Work of the API Project 6 on the Hydrocarbons in the $C_{13}$ to $C_{38}$ Fraction of Petroleum—Beveridge J. Mair and Frederick D. Rossini
Determination of the Paraffin, Monocyclic and Dicyclic Napthene Contents of a Middle East Gas Oil by Selective Adsorption—H. C. Rampton
Composition of an East Texas Lubricating Oil Distillate—B. A. Orkin, J. G. Bendoraitis, B. Brown, and R. H. Williams
New Thermal Diffusion Techniques Applicable to High-Boiling Petroleum Fractions—A. L. Jones
The Separation of High-Boiling Petroleum Oils by Thermal Diffusion—F. W. Melpolder, R. W. Sauer, and T. A. Washall
Solution Behavior Patterns as Supplement in Analysis of Oil Fractions—E. L. Derr, C. H. Deal, and G. J. Pierotti

### Spectroscopic Methods

Introduction—E. J. Rosenbaum
Determination of Methyl, Methylene, and Alkyl Benzene Group Types by Infrared Absorption—R. H. Hughes and R. J. Martin
Analysis of Petroleum Oils by Mass Spectrometry—E. G. Carlson and M. J. O'Neal
Characterization of Hydrocarbons in Petroleum by Nuclear Magnetic Resonance Spectrometry—R. B. Williams

The Influence of Intermolecular Interactions on Nuclear Magnetic Resonance Chemical Shift Measurements as Related to Certain High Molecular Weight Hydrocarbon Systems—J. R. Zimmerman and J. A. Lasater

### General Methods and Correlations

Introduction—R. L. Le Tourneau
The Composition of Petroleum Distillates as Revealed by Their Sulfonates—A. B. Brown and J. O. Knobloch
Adsorption and Physical Properties Methods—W. J. Stout, R. W. King, M. E. Peterkin, and S. S. Kurtz, Jr.
Comparison of Structural Group Analyses by Spectrometric and Physical Property Methods—S. H. Hastings, B. H. Johnson, H. E. Lumpkin, and R. B. Williams
Molecular Weight Determination—F. H. Stross
A Correlative Method for Structure of Solid Saturated Hydrocarbons—G. G. Rumberger and R. W. Dannenbrink

The papers with discussion were issued as *ASTM Special Technical Publication No. 224* entitled "Symposium on Composion of Petroleum Oils."

# 1957 TECHNICAL PAPERS PUBLISHED IN ASTM BULLETIN

## ASTM Bulletin *No. 219, January 1957*

Increasing Integration in the Manufacture of Forest Products in the Northwest—O. H. Schrader, Jr., p. 25

A Mechanism to Operate a Tension Tester at Constant Strain Rates—Stephen Strella, p. 29 (TP 1)

Measurement of the Elastic Modulus of Films—J. R. Olson and D. W. Vanas, p. 34 (TP 6)

Thermal Coefficient of Expansion of Concrete Aggregates—William Lerch, p. 36 (TP 8)

Statistical Quality Control in a Petroleum Control Laboratory—J. M. Duncan, p. 40 (TP 12)

Solvent Degreasing—What Every User Should Know—C. E. Kircher, p. 44 (TP 16); Disc., p. 49 (TP 21)

Determination of Water-Soluble Soil Removal in Drycleaning—G. P. Fulton, A. C. Lloyd, F. Loibl, Gloria Moore, and Doris Reichard, p. 50 (TP 22)

## ASTM Bulletin *No. 220, February 1957*

Use Specifications in Buying Rubber Parts—R. B. Stringfield, p. 39 (TP 27)

Asphalt Research at the National Bureau of Standards—H. R. Snoke, p. 41 (TP 29)

Water-Soluble Degradation Products of Asphalt—Alan Schriesheim and S. Greenfeld, p. 43 (TP 31)

Production and Fabrication of New AISI Types 201 and 202—G. W. Hinkle, p. 47 (TP 35)

New Method for Recording Explosion Impulse Effects on Solids—W. E. Bron, K. A. Zadwick, and A. L. Tarr, p. 50 (TP 38)

The Impact-Absorbing Capacity of Textile Yarns—J. C. Smith, F. L. McCrackin, and H. F. Scheifer, p. 52 (TP 40)

Measurement of Cure Rate of Thermosetting Molding Materials—A. J. Guzzetti, p. 57 (TP 45)

Accelerated Fatigue of Plastics—L. S. Lazar, p. 67 (TP 55)

## ASTM Bulletin *No. 221, April 1957*

Protective Coating Adhesion Measurement Using an Electronic Averaging Device for the Adherometer—A. G. Roberts and R. S. Pizer, p. 53 (TP 61)

The Systematic Analysis of Deposits from Oil-Fired Furnaces—J. W. McCoy, p. 59 (TP 67)

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Production and Fabrication of New AISI Types 201 and 202. Published in ASTM BULLETIN, No. 220, Feb., 47 (TP35).

**Hodge, John M.**
Discussion, 713.

**Hoff, E. A. W., and Turner, S.**
A Study of the Low-Temperature Brittleness Testing of Polyethylene. Published in ASTM BULLETIN, No. 225, Oct., 58 (TP208).

**Hoke, J., Eberle, F., and Wylie, R. D.**
Embrittling Tendencies of Austenitic Superheater Materials at Elevated Temperatures, 821. Disc., 832.

**Holloway, J. M.**
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**Holt, K. E.**
Results of Collaborative Viscosity Tests—Report of Subcommittee IX, of Committee D-1, Group 18, 297.

**Holtz, W. G.**
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**Horger, O. J., and Neifert, H. R.**
Fretting Corrosion of Large Shafts as Influenced by Surface Treatments—Summary of Proceedings of *STP 216*, 698.

**Horne, G. T.**
See Lipsitt, H. A., and Horne, G. T.

**Housel, William S.**
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**Hoyt, S. L.**
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**Hueschen, R. E.**
See Kelly, W. S., Kaulitz, D. C., and Hueschen, R. E.

**Hughes, F. J., and Walker, D. C.**
Gloss and Gloss Stability of Waxed Papers—Summary of Proceedings of Symposium on Wax, 1370.

**Hughes, Richard H., and Martin, Robert J.**
Determination of Methyl, Methylene, and Alkyl Benzene Group Types of Infrared Absorption—Summary of Proceedings of *STP 224*, 1374.

**Hunter, Thomas A.**
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**Hunter, M. S., and Fricke, Wm. G., Jr.**
Cracking of Notch Fatigue Specimens, 643 (*R*). Disc., 653.

**Hyler, W. S.**
See Tarasov, L. P., Hyler, W. S., and Letner, H. R.

J

**Jackman, K. R.**
Discussion. Published in ASTM BULLETIN, No. 220, Feb., 40 (TP28).

**Jacquet, Pierre A.**
Nondestructive Technique for Macro and Micrographic Surface Examination of Metallic Specimens (Electrolytic Local Polishing and Replica Technique), 1290. Disc., 1303.

**Johnson, B. H.**
See Hastings, S. H., Johnson, B. H., Lumpkin, H. E., and Williams, R. B.

**Jones, A. Letcher**
New Thermal Diffusion Techniques Applicable to High Boiling Petroleum Fractions—Summary of Proceedings of *STP 224*, 1374.

**Jones, M. H., Shannon, Jr., J. L., and Brown, Jr., W. F.**
Influence of Notch Preparation and Eccentricity of Loading on the Notch Rupture Life, 833 (*R*). Disc., 850.

**Joy, F. A.**
Thermal Conductivity of Insulation Containing Moisture—Summary of Proceedings of Philadelphia Spring Meeting, *33*.

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**Kallman, Silve**
See Codell, M., Norwitz, G., and Kallman, Silve.

**Karol, R. H.**
Development of a New Chemical Grout, 1219. Disc., 1231.

**Kattus, J. R.**
Properties of Cast Iron at Elevated Temperatures. Published in ASTM BULLETIN, No. 222, May, 12.

**Katz. H. L., Proctor, K. L., and Nagley, F.**
Performance of Various Chromate Supplementary Films on Electrodeposited Zinc Coatings, Report of Committee B-8 (appendix), 203.

**Kaufman, J. G.**
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**Kaulitz, D. C.**
See Kelly, W. S., Kaulitz, D. C., and Hueschen, R. E.

**Kelly, T. M., and Bryant, D. E.**
Measuring the Rate of Hardening of Concrete by Bond Pullout Pins, 1029. Disc., 1041.

**Kelly, W. S., Kaulitz, D. C., and Hueschen, R. E.**
Techniques of Tension Testing Irradiated Materials at Elevated Temperatures at Hanford—Summary of Proceedings of *STP 220*, 1368.

**Kesler, C. E.**
See Murdock, J. W., and Kesler, C. E.

**Kesler, C. E., and Chang, T. S.**
A Review of Sonic Methods for the Determination of Mechanical Properties of Solid Materials. Published in ASTM BULLETIN, No. 225, Oct., 40 (TP190). (*R*).

**King, R. W.**
See Stout, W. J., King, R. W., Peterkin, M. E., and Kurtz, S. S.

**Kircher, C. E.**
Solvent Degreasing—What Every User Should Know. Published in ASTM BULLETIN, No. 219, Jan., 44 (TP16). (*R*) Disc., 49 (TP21).

**Klett, Roy A.**
General Introduction to Symposium on Composition of Petroleum Oils—Summary of Proceedings of *STP 224*, 1374.

**Klier, E. P.**
See Muvdi, B. B., Sachs, G., and Klier, E. P.

**Klier, E. P., Muvdi, B. B., and Sachs, G.**
The Static Properties of Several High-Strength Steels (*R*), 715. Disc., 728.

**Knobloch, J. O.**
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**Koerner, W. E.**
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**Kutz, Myer P.**
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L

**Lacks, Hyman, Quatinetz, Max, and Freiberger, Arnold**
Ice Adhesion Apparatus and Test Method. Published in ASTM BULLETIN, No. 224, Sept., 48 (TP168).

**Lampson, F. K., Tsareff, Jr., T. C., and Green, A. W. F.**
Thermal Shock Testing Under Stress of Certain High Temperature Alloys, 965.

**Lasater, J. A.**
See Zimmerman, J. R., and Lasater, J. A.

**Lazar, L. S.**
Accelerated Fatigue of Plastics. Published in ASTM BULLETIN, No. 220, Feb., 67 (TP55).

**Leep, R. W.**
See Ross, J. D., and Leep, R. W.

**Leeser, D. O.**
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See Calkins, G. D., Leeser, D. O., Gates, J. E., Rough, F. A., and DelGrosso, A.

**Lerch, William**
Thermal Coefficient of Expansion of Concrete Aggregates. Published in ASTM BULLETIN, No. 219, Jan., 36 (TP8). (*R*).

**Lessells, J. M.**
Introduction to Symposium on Large Fatigue Testing Machines and Their Results—Summary of Proceedings of *STP 216*, 698.

**Letner, H. R.**
See Tarasov, L. P., Hyler, W. S., and Letner, H. R.

**Leverenz, E. G.**
See Bradley, G. E., Leverenz, E. G., and McGonnagle, W. G.

**Libby, H. L.**
Introduction to Eddy Current Methods and Techniques—Summary of Proceedings of *STP 223*, 1372.
See Linsey, G. D., and Libby, H. L.

**Linsey, G. D., and Libby, H. L.**
A Nickel Depth Meter—Summary of Proceedings of *STP 223*, 1372.

**Lipsitt, H. A., and Horne, G. T.**
The Fatigue Behavior of Decarburized Steel, 587 (*R*). Disc., 600.

**Litehiser, R. R.**
Discussion. Published in ASTM BULLETIN, No. 222, May, 34 (TP102).

**Liu, H. W., Corten, H. T., and Sinclair, G. M.**
Fretting Fatigue Strength of Titanium Alloy RC 130B, 623 (*R*). Disc., 642.

**Lloyd, A. C.**
See Fulton, G. P., Lloyd, A. C., Loibl, F., Moore, Gloria, and Reichard, Doris.

**Loibl, F.**
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**Lubahn, J. D.**
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**Lumpkin, H. E.**
See Hastings, S. H., Johnson, B. H., Lumpkin, H. E., and Williams, R. B.

**Lumsden, G. Q.**
Wood Poles for Communication Lines. Published in ASTM BULLETIN, No. 222, May, 19 (TP87). (*R*). Disc., 24 (TP92).

**Lusk, E. C.**
See Calkins, G. D., Whitney, J. E., and Lusk, E. C.

**Lustman, B.**
Engineering Effects of Radiation on Nuclear Fuels—Summary of Proceedings of *STP 220*, 1368.

**Lutz, W. H.**
ACR Notes. Published in ASTM BULLETIN, No. 220, Feb., 17.

M

**Mair, Beveridge J., and Rossini, Frederick D.**
Summary of Work of the API Research Project 6 on Hydrocarbons in the $C_{13}$ to $C_{38}$ Fraction of Petroleum—Summary of Proceedings of *STP 224*, 1374.

**Manjoine, M. J.**
Combination Creep-Rupture Test Specimen. Published in ASTM BULLETIN, No. 222, May, 36 (TP104).
Discussion, 1272.

**Manjoine, M. J., Wessel, E. T., and Pryle, W. H.**
Constant Head Rate Testing Machine with Instantaneous Speed Change. Published in ASTM BULLETIN, No. 226, Dec., 31 (TP219). (*R*). Disc., 35 (TP223).

**Mannal, C.**
See Plant, W. R., and Mannal, C.

**Marble, J. D.**
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**Marburger, W. G., Monaweck, J. H., and McGonnagle, W. J.**
Electrode Potential Method of Bond Testing—Summary of Proceedings of *STP 223*, 1372.

**Marin, Joseph**
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**Markwardt, L. J.**
Discussion. Published in ASTM BULLETIN, No. 222, May, 24 (TP92).

**Martin, David E.**
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**Martin, Robert J.**
See Hughes, Richard H., and Martin, Robert J.

**Mather, B.**
Discussion. Published in ASTM BULLETIN, No. 226, Dec., 47 (TP235); No. 226, Dec., 51 (TP239).

**Matters, Robert**
Discussion, 945.

**Mattimore, J. D.**
Lightweight Corrosion-Resistant Pipe Standards Make Progress. Published in ASTM BULLETIN, No. 221, April, 45.

**Mattson, R. L.**
Discussion, 620.

**Matyas, Eugene**
Discussion, 1231.

**Mavis, F. T., and Greaves, M. J.**
Instrumentation and Method for Impulse Testing of Reinforced-Concrete Beams. Published in ASTM BULLETIN, No. 226, Dec., 43 (TP231). Disc., 47 (TP235).

**Maxon, G.**
Discussion. Published in ASTM BULLETIN, No. 226, Dec., 47 (TP235).

**Maxon, Glenway**
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**McBurney, J. W.**
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**McClintock, Frank A.**
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**McClung, R. W.**
See Oliver, R. B., McClung, R. W., and White, J. K.

**McConnell, T. A.**
Evaluation of Laboratory Tests as Indicators of the Service Life of Uninhibited Electrical Insulating Oils—Summary of Proceedings of *STP 218*, 1373.

**McCoy, J. W.**
The Systematic Analysis of Deposits from Oil-Fired Furnaces. Published in ASTM BULLETIN, No. 221, April, 59 (TP67).

**McCrackin, F. L.**
See Smith, J. C., McCrackin, F. L., and Schiefer, H. F.

**McGonnagle, W. J.**
Introduction to Symposium on Nondestructive Tests in the Field of Nuclear Energy—Summary of Proceedings of *STP 223*, 1371.
See Bradley, G. E., Leverenz, E. G., and McGonnagle, W. J.
See Bradley, G. E., McGonnagle, W. J., and Gonzales, P. R.
See Marburger, W. G., Monaweck, J. H., and McGonnagle, W. J.
See Monaweck, J. H., McGonnagle, W. J.
See Renken, Jr., C. J., and McGonnagle, W. J.
Testing of Cylindrical Fuel Elements Using the Cyclograph—Summary of Proceedings of *STP 223*, 1372.

**McLain, Stuart**
Nondestructive Testing in the Nuclear Energy Field—Summary of Proceedings of *STP 223*, 1371.

**Melpolder, F. W., Sauer, R. W., and Washall, T. A.**
The Separation of High Boiling Petroleum Oils by Thermal Diffusion—Summary of Proceedings of *STP 224*, 1374.

**Menzel, Carl A.**
Fallacies in the Current per Cent of Total Absorption Method for Determining and Limiting the Moisture Content of Concrete Block, 1057. Disc., 1072.

**Michel, R.**
Influence of Operating Experience and Full Scale Tests on Propulsion Shafting Design of U. S. Navy Ships—Summary of Proceedings of *STP 216*, 698.

**Miller, D. G.**
Development of Drain Tile Specifications in ASTM—A Brief History. Published in ASTM BULLETIN, No. 221, April, 47.

**Mitchell, Nolan D.**
Discussion, 1041.

**Molander, B. L., Waldron, C. R., and Newland, J. C.**
A Fixture for Compression Testing of Sheet Materials at Elevated Temperatures. Published in ASTM BULLETIN, No. 225, Oct., 37 (TP187).

**Monaweck, J. H.**
See Marburger, W. G., Monaweck, J. H., and McGonnagle, W. J.

**Monaweck, J. H., and McGonnagle, W. J.**
Thermal Testing of Reactor Fuel Elements—Summary of Proceedings of *STP 223*, 1372.

**Moore, Eugene B.**
Discussion. Published in ASTM BULLETIN, No. 222, May, 35 (TP103).

**Moore, Gloria**
See Fulton, G. P., Lloyd, A. C., Loibl, F., Moore, Gloria, and Reichard, Doris.

**Morgan, J. R.**
Hanford's Improved Remote Metallographic Equipment—Summary of Proceedings of *STP 220*, 1368.

**Morris, P. R.**
See Scheuer, J. P., and Morris, P. R.

**Morris, J. M., and Pink, F. X.**
Trace Analysis by Means of the Graphite Spark—Summary of Proceedings of *STP 221*, 1367.

**Morris, R. E., and Holloway, J. M.**
Evaluation of a Microhardness Tester. Published in ASTM BULLETIN, No. 222, May, 45 (TP113).

**Morrow, Jo-Dean**
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**Munch, R. H.**
See Ashworth, G. W., Darby, J. R., Koerner, W. E., and Munch, R. H.

**Murakawa, Kiyoshi**
See Richards, John T., and Murakawa, Kiyoshi.

**Murata, K. J.**
Geological Materials—Summary of Proceedings of *STP 221*, 1367.

**Murdock, J. W., and Kesler, C. E.**
Effect of Length to Diameter Ratio of Specimen on the Apparent Compressive Strength of Concrete. Published in ASTM BULLETIN, No. 221, April, 68 (TP76).

**Murphey, R. L., and Chandler, Jr., W. B.**
Effect of Refining Conditions on Wax Quality—Summary of Proceedings of Symposium on Wax, 1370.

**Murphy, R. B.**
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**Muscatell, F. L., Reynolds, E. E., Dyrkacz, W. W., and Dalheim, J. H.**
Thermal Shock Resistance of High Temperature Alloys, 947. Disc., 963.

**Muvdi, B. B.**
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**Nance, R. A.**
See Allen, J. W., Nance, R. A., and Oliver, R. B.

**Neifert, H. R.**
See Horger, O. J., and Neifert, H. R.

**Nelson, S.**
See Xavier, M. A., Rotariu, G. J., Nelson, S., Yefsky, A., and Walters, A.

**Nevill, Gale E., Jr., and Brotzen, Franz, R.**
The Effect of Vibrations on the Static Yield Strength of a Low-Carbon Steel, 751 (*R*).

**Newland, J. C.**
See Molander, B. L., Waldron, C. R., and Newland, J. C.

**Norris, J. A.**
Spectrographic Determination of Trace Elements in Metals—Summary of Proceedings of *STP 221*, 1367.

**Norwitz, G.**
See Codell, M., Norwitz, G., and Kallman, Silve

O

**Oakes, A. E.**
Radiographic Inspection of Nuclear Core Materials and Components—Summary of Proceedings of *STP 223*, 1372.

**Okerson, W. E.**
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**Oliver, R. B.**
See Allen, J. W., Nance, R. A., and Oliver, R. B.
See Allen, J. W. and Oliver, R. B.

**Oliver, R. B., McClung, R. W., and White, J. K.**
Production Inspection of Pipe and Tubing by the Immersed Ultrasonic Method—Summary of Proceedings of *STP 223*, 1371.

**Oliver, R. B., Tolson, G. M., and Taboada, A.**
The Use of Penetrants for Inspection of Small Diameter Tubing—Summary of Proceedings of *STP 223*, 1371.

**Olsen, Harold W.**
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**Olson, J. R., and Vanas, D. W.**
Measurement of the Elastic Modulus of Films. Published in ASTM BULLETIN, No. 219, Jan., 34 (TP6).

**O'Neal, M. J.**
See Carlson, E. G. and O'Neal, M. J.

**Orkin, B. A., Bendoraitis, J. G., Brown, B. and Williams, R. H.**
Composition of an East Texas Lube Oil Distillate—Summary of Proceedings of *STP 224*, 1374.

**Otto, W. C.**
A Statistical Study of Flexural Strength of Concrete Beams, •1154. Disc., 1168.

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**Palmer, L. A.**
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**Pappin, W. H.**
Helium Leak Detection Techniques—Summary of Proceedings of *STP 223*, 1372.

**Partridge, Everett, P.**
Your Most Important Raw Material (Marburg Lecture). Summary of Proceedings 23, 49 (issued as separate pamphlet).

**Peterkin, M. E.**
See Stout, W. J., King, R. W., Peterkin, M. E., and Kurtz, S. S.

**Petersen, P. H.**
Discussion. Published in ASTM BULLETIN, No. 226, Dec., 47 (TP235).

**Peterson, W. S.**
Discussion. Published in ASTM BULLETIN, No. 226, Dec., 42 (TP230).

**Petry, W. R.**
Discussion. Published in ASTM BULLETIN, No. 219, Jan., 49 (TP21).

**Pevar, Maxwell**
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**Philleo, R. E.**
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**Phillips, Joseph**
Factors Influencing the Staining Tendency of Wax—Summary of Proceedings of Symposium on Wax, 1370.

**Pierotti, G. J.**
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**Pink, F. X.**
See Morris, J. M., Pink, F. X.

**Pirtz, D.**
See Best, C. H., Pirtz, D., and Polivka, M.

**Pizer, R. S.**
See Roberts, A. G., and Pizer, R. S.

**Plant, W. R.**
A Recording X-ray Photometer—Summary of Proceedings of *STP 223*, 1372.

**Plant, W. R. and Mannal, C.**
An Eddy Current Test for Capillary Tubing—Summary of Proceedings of *STP 223*, 1371.

**Podereskin, M.**
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**Polivka, M.**
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**Price, R. B.**
See Calkins, G. D., Podereskin, M., Price, R. B., and Ewbank, Jr., N. M.

**Price, W. H.**
Discussion. Published in ASTM BULLETIN, No. 226, Dec., 47 (TP235).

**Proctor, K. L.**
See Katz, H. L., Proctor, K. L., and Nagley, F.

**Proudley, Charles E., and Waller, H. Fred, Jr.**
A Cooperative Investigation of the Method of Test for Compressive Strength of Bituminous Mixtures, 1321.

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Tensile and Creep Properties of Tungsten at Elevated Temperatures, 906. Disc., 916.

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**Quatinetz, Max**
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**Raab, Edward L.**
The Use of Solid and Liquid Catalysts for the Accelerated Aging Testing of Transformer Oil—Summary of Proceedings of *STP 218*, 1373.

**Rampton, H. C.**
Determination of the Paraffin, Monocyclic and Dicyclic Naphthene Contents of a Middle East Gas Oil by Selective Adsorption—Summary of Proceedings of *STP 224*, 1374.

**Randall, P. M.**
Constant-Stress Creep Rupture Tests of a Killed Carbon Steel, 854. Disc. 871.

**Reichard, Doris**
See Fulton, G. P., Lloyd, A. C., Loibl, F. Moore, Gloria, and Reichard, Doris.

**Renken, Jr., C. J. and McGonnagle, W. J.**
Eddy Current Techniques for Testing Liquid Metal Bonding—Summary of Proceedings of *STP 223*, 1372.

**Renken, Jr., C. J. and Waidelich, D. L.**
Minimizing the Effect of Probe-to-Metal Spacing in Eddy Current Testing—Summary of Proceedings of *STP 223*, 1372

**Reynolds, E. E.**
See Muscatell, F. L., Reynolds, E. E., Dyrkacz, W. W., and Dalheim, J. H.

**Rhoten, Merle L.**
Radiography of Fuel Elements and Fuel Materials Using Cesium-137—Summary of Proceedings of *STP 223*, 1372.

**Rich, P. J.**
Discussion. Published in ASTM BULLETIN, No. 219, Jan., 49 (TP21).

**Richards, J. T.**
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**Richards, John T. and Murakawa, Kiyoshi**
Effect of Cold Rolling and Heat Treatment on the Directional Properties of Beryllium Copper Strip, 791 (*R*).

**Richards, John T. and Smith, E. M.**
Problems Associated with Hardness Conversion of Several Copper Alloys. Report of Committee B-5 (appendix), 161.

**Ridenour, W. P., Spilners, I. J., and Templin, P. R.**
Composition *versus* Properties of Microcrystalline Waxes—Summary of Proceedings of Symposium on Wax, 1370.

**Rider, D. K.**
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**Ristaino, A. J., and Dominick, A. A.**
Evaluation of Hartmann and Braun Dissolved Oxygen Recorder for Boiler Feedwater—Summary of Proceedings of *STP 219*, 1369.

**Roberts, A. G., and Pizer, R. S.**
Protective Coating Adhesion Measurement Using an Electronic Averaging Device for the Adherometer. Published in ASTM BULLETIN, No. 221, April, 53 (TP61).

**Roberts, J. G.**
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**Robinson, Henry E.**
Introduction to Symposium on Thermal Conductivity Measurements and Applications of Thermal Insulations—Summary of Proceedings of Philadelphia Spring Meeting, 33.

**Robinson, R. C.**
A Nondestructive Test for Intergranular Corrosion in Stainless Steel—Summary of Proceedings of *STP 223*, 1371.

**Roden, H.**
Controlled Moisture Condensation Apparatus for Evaluation of Rust-Preventive Oils. Published in ASTM BULLETIN, No. 223, July, 55 (TP121).

**Rosard, D. D.**
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**Rosenbaum, E. J.**
Introduction to Section II of Symposium on Composition of Petroleum Oils—Summary of Proceedings of *STP 224*, 1374.

**Rosenberg, Samuel J.**
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**Ross, J. D. and Leep, R. W.**
Ultrasonic Transmission Tester for Detection of Unbonded Areas—Summary of Proceedings of *STP 223*, 1372.

**Ross, S. T. and Teague, D. M.**
The Preparation of Shadowed Plastic Replicas, Report of Committee E-4 (appendix), 527.

**Rossini, Frederick D.**
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**Rotariu, G. J.**
See Xavier, M. A., Rotariu, G. J., Nelson, S., Yefsky, A., and Walters, A.

**Rough, F. A.**
See Calkins, G. D., Leeser, D. O., Gates, J. E., Rough, F. A., and DelGrosso, A.

**Rumberger, G. G.**
The Effect of Paper-Making Materials on Wax Oxidation Rates—Summary of Proceedings of Symposium on Wax, 1370.

**Rumberger, G. G. and Dannenbrink, R. W.**
A Correlative Method for Structure of Solid Saturated Hydrocarbons—Summary of Proceedings of *STP 224*, 1375.

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**Sachs, G.**
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**Sachs, George and Scheven, G.**
Relation Between Direct-Stress and Bending Fatigue of High-Strength Steels, 667 (*R*).

**Sauber, W. J.**
Discussion. Published in ASTM BULLETIN, No. 225, Oct., 52 (TP202).

**Sauer, R. W.**
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**Sawyer, James L.**
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**Saxer, E. L., and Toennies, H. T.**
Measuring Shrinkage of Concrete Block, 988. Disc., 1006.

**Schatzel, R. A.**
Our Expanding Technology and ASTM—1957 President's Address, 34. Published in ASTM BULLETIN, No. 223, July, 53 (*also* published as separate pamphlet).

**Schelleng, R. D. and Eash, J. T.**
Effect of Composition on the HighTemperature Properties of Ductile Iron, 929 (*R*). Disc., 945.

**Scheuer, J. P. and Morris, P. R.**
Measurement of Density of Uranium Metal by Radiation Absorption—Summary of Proceedings of *STP 223*, 1372.

**Scheven, G.**
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**Scheven, G., Sachs, G., and Tong, K.**
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**Schiefer, H. F.**
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**Schlabach, T. D., Wright, E. E., Broyer, A. P., and Rider, D. K.**
Testing of Foil-Clad Laminates for Printed Circuitry. Published in ASTM BULLETIN, No. 222, May, 25 (TP93).

**Schmid, Werner E.**
The Permeability of Soils and the Concept of a Stationary Boundary-Layer, 1195 (*R*). Disc., 1211.

**Schofield, L. B.**
Introduction to Symposium on Insulating Oils—Summary of Proceedings of *STP 218*, 1373.

**Schrader, Jr., O. H.**
Increasing Integration in the Manufacture of Forest Products in the Northwest. Published in ASTM BULLETIN, No. 219, Jan., 25.

**Schrenk, W. G.**
Plants and Soils—Summary of Proceedings of *STP 221*, 1367.

**Schriesheim, Alan, and Greenfeld, S. H.**
Water-Soluble Degradation Products of Asphalt. Published in ASTM BULLETIN, No. 220, Feb., 43 (TP31). (*R*).

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      laboratory and service tests of marine shafting (Bunyan).
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      sudden fracture of machine parts and structure elements (Uzhik, Galperin, and Zooykova).
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  of steel stressed in torsion (Hendrickson). Published in ASTM BULLETIN, No. 224, Sept., 40 (TP160).
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- Symposium. Summary of Proceedings of *STP 223*, 1371:
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    - eddy current
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      - fuel element cladding (Allen, Nance, and Oliver).
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- Nuclear Power in a Package. Published in ASTM BULLETIN, No. 224, Sept., 9.
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  - the California story (Vesper). Published in ASTM BULLETIN, No. 220, Feb., 21.
  - *See* Insulating Oils
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  - California oil story (Vesper). Published in ASTM BULLETIN, No. 220, Feb., 21.
  - quality control laboratory (Duncan). Published in ASTM BULLETIN, No. 219, Jan., 40 (TP12).
  - Symposium on Composition of Petroleum Oils. Summary of Proceedings of *STP 224*, 1374:
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      - molecular weight (Stross).
      - solid saturated hydrocarbons (Rumberger and Dannenbrink).
      - spectrometric and physical property methods (Hastings, Johnson, Lumpkin, and Williams).
    - introduction (Klett).
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    - separation
      - API Project 6 on hydrocarbons in $C_{13}$ to $C_{38}$ fraction (Mair and Rossini).
      - East Texas lubricating oil distillate (Orkin, Bendoraitis, Brown, and Williams).
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Progress Report
insulating materials
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      organic materials as reactor coolant-moderators (Colichman and Gercke).
      radiation stability of hydrocarbon fuels (Carroll, Bolt, and Bert).
      radiation stability of 10 per cent by weight molybdenum-uranium fuel alloys (Calkins, Leeser, Gates, Rough, and DelGrosso).
      standardization of techniques in radiation studies (Feldman and Fillnow).
      structural materials for nuclear power plants (Fleischmann).
      tension testing irradiated materials at Hanford (Kelly, Kaulitz, and Hueschen).
      visual examinations and dimensional measurements at Hanford (Zimmerman).
Radioactive Materials
  corrosion and heat-treatment (Calkins, Whitney, and Lusk). Published in ASTM BULLETIN, No. 224, Sept., 25 (TP145).
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- Sorptive Mineral Materials
  - new ASTM committee organized. Published in ASTM BULLETIN, No. 222, April, 35.
- Specifications
  - sampling plans in ASTM specifications. Published in ASTM BULLETIN, No. 223, July, 47.
  - drain tile (Miller), Published in ASTM BULLETIN, No. 221, April, 47.
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  - for trace elements
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      - by means of graphite spark (Morris and Pink).
      - determination in metals
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      - in materials
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        - medical and biological (Vallee).
      - in plants and soils (Schrenck).
      - introduction (Timma).
  - nickel alloys
    - powder d-c arc technique, proposed method.
      - Report of Committee E-2 (appendix), 436.
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    - Symposium on Composition of Petroleum Oils. Summary of Proceedings of *STP 224*, 1374:
      - infrared absorption (Hughes and Martin).
      - introduction (Rosenbaum).
      - mass spectrometry (Carlson and O'Neal).
      - nuclear magnetic resonance
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  - Report of Administrative Comm. on Standards, 66.
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- Symposium on Large Fatigue Testing Machines and Their Results. Summary of Proceedings of *STP 216*, 698:
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  - torsion testing of axle shafts (Eckert).
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# 1957 LIST OF SPECIAL TECHNICAL PUBLICATIONS

The publications listed below have been compiled during the year 1957 to be issued as separate publications not included in either *Proceedings* or ASTM BULLETIN. For a list of earlier Special Technical Publications, see the original listings in Proceedings, Volume 48, page 1352, and the supplementary listings in Volume 49 page 1240, Volume 50, page 1490, Volume 51, page 1322, Volume 52, page 1309, Volume 53, page 1197, Volume 54, page 1388, Volume 55, page 1250, and Volume 56, page 1498. A complete list of Special Technical Publications covering all publications, up to and including 1950, is included in the 50 Year Index to ASTM Publications. Also a complete list up to and including 1955 is published in the Five-Year Index to ASTM Publications (1951–1955).

| SPECIAL TECHNICAL PUBLICATION NUMBER | TITLE |
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| 56-K | 1956 Supplement to Bibliography and Abstracts on Electrical Contacts (1957) |
| 90-D | 1957 Supplement to the Metal Cleaning Bibliography |
| 150-B | 1957 Supplement to Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents |
| 170-A | Compilation of Chemical Composition and Rupture Strengths of Super-Strength Alloys (1957) |
| 203 | *Aircraft Fatigue Papers (1956) |
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| 217 | Symposium on Thermal Insulating Materials (1957) |
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| 219 | Symposium on Determination of Dissolved Oxygen in Water (1957) |
| 220 | Symposium on Radiation Effects on Materials—Vol. 2 (1957) |
| 221 | Symposium on Spectrochemical Analysis for Trace Elements (1957) |
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| 223 | Symposium on Nondestructive Tests in the Field of Nuclear Energy (1957) |
| 224 | Symposium on Composition of Petroleum Oils (1957) |
| 225 | Knocking Characteristics of Hydrocarbons (1957) |
| 226 | Symposium on Composition of Elevated Temperature Properties of Weld Deposits (1957) |

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*West Coast Paper.